The Journal
of the
Society of Dyers and Colourists

CONTENTS

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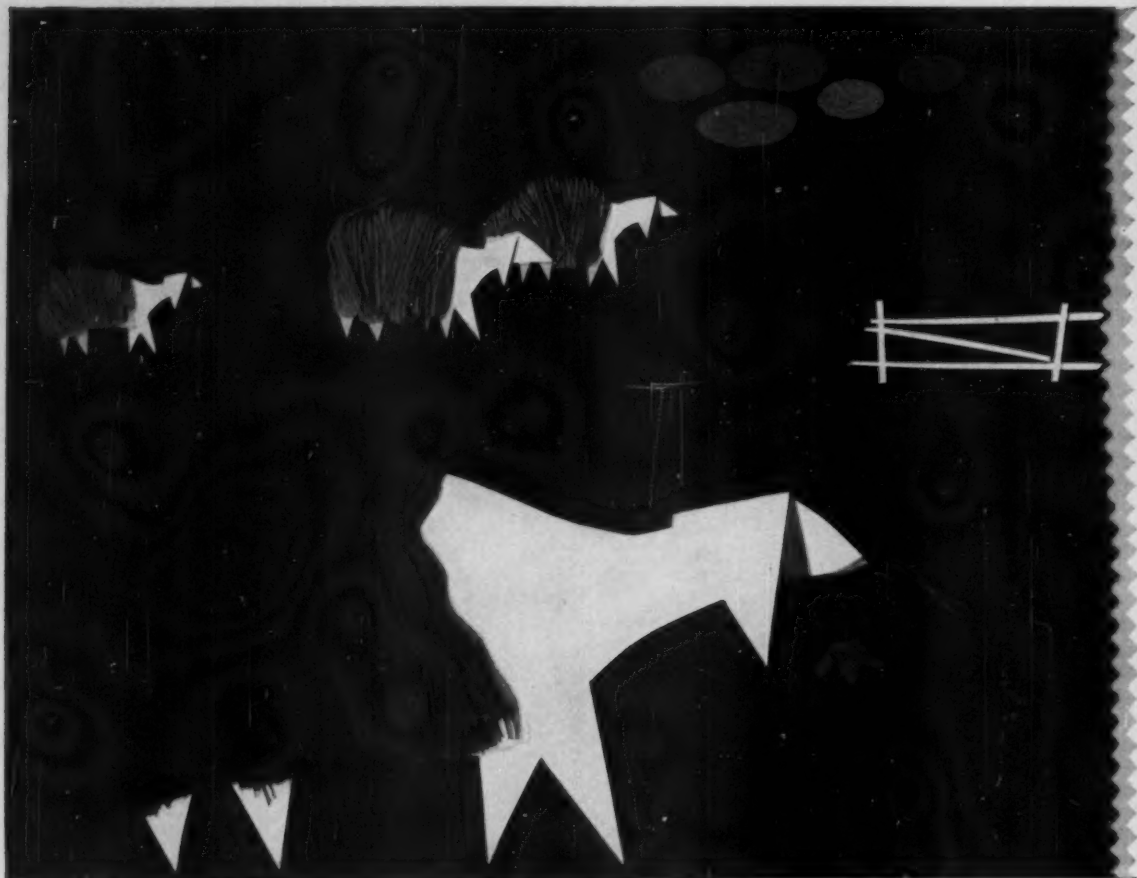1954 1955 1956 1957 1958 1959 Price 1s each

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The Journal of the Society of Dyers and Colourists

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Legal and Practical Problems of Effluent Disposal   *J. H. Harwood*

Combined Dyeing and Finishing of Cellulosic Piece Goods   *I. D. Rattee*

COMMUNICATIONS

Cross-linking of Cellulose and its Derivatives

     I— Use of Unsaturated Esters in Cross-linking Reactions   *W. M. Corbett and J. E. McKay*

     VI— Addition of Amines to Cellulose Crotonates   *J. E. McKay and W. Taylor*

Fibre Surface Contaminants and their Effect in Shrinkproofing   *A. J. Farnworth*

Shrinkproofing Wool with Reducing Agents   *A. J. Farnworth*

Further Tests for Colour Vision   *F. Jordinson*

Photochemistry of Sodium Chlorite in Solution, with special reference to Chlorite Bleaching   *C. Kujirai and I. Fujita*

Characterisation of Solvent-soluble Dyes by Optical Transmission Methods   *I. F. Trotter*

The Formation of Polymers in Wool   *L. J. Wolfram and J. B. Speakman*

Announcement

FORTHCOMING MEETINGS OF THE SOCIETY

This *announcement* is to inform all members of the discontinuance of the publication of 'Forthcoming Meetings' in the Society's *Journal*. Each Section will have a separate booklet giving their meetings and a small number of these will be available to each of the other Sectional Honorary Secretaries and on application for visiting members from Overseas.
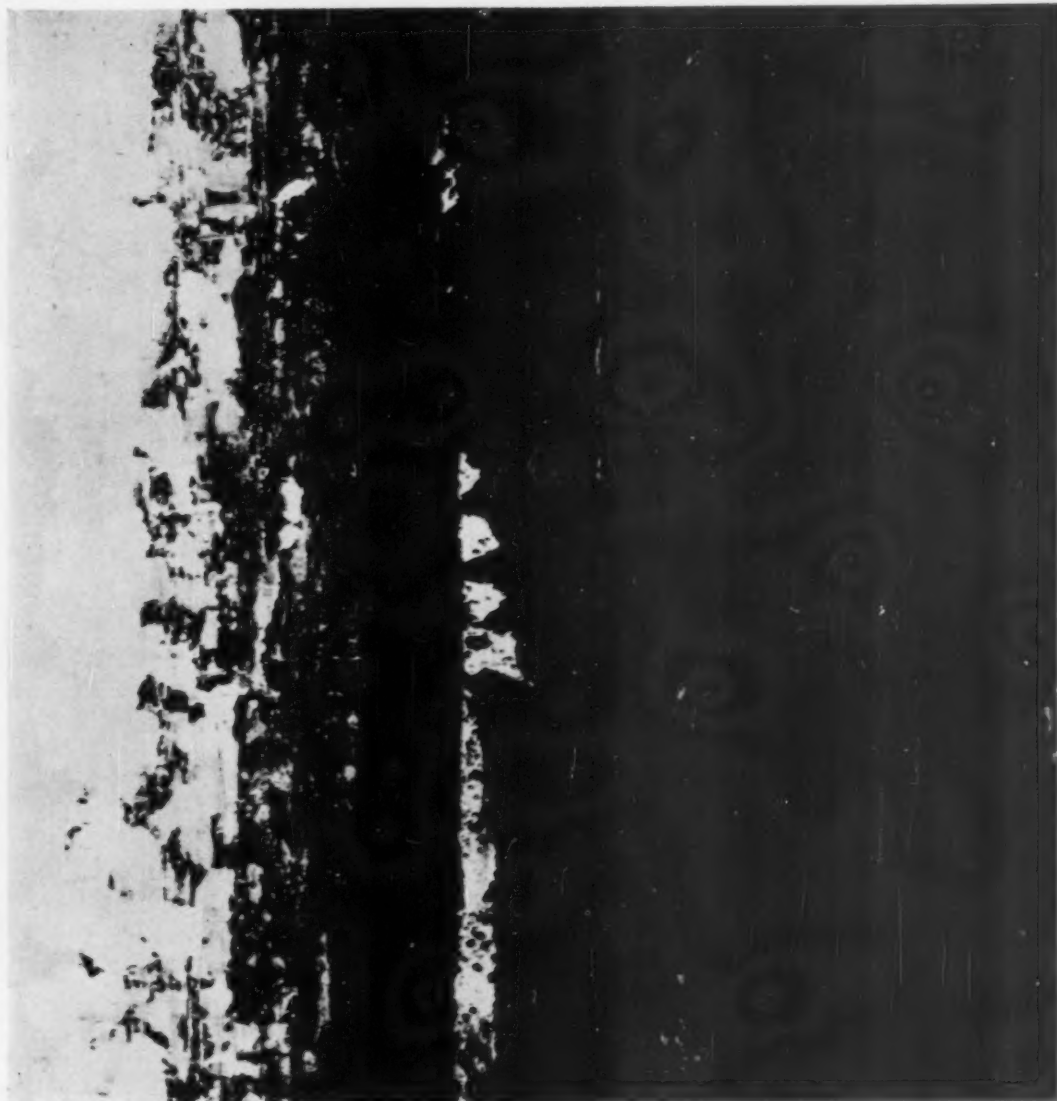
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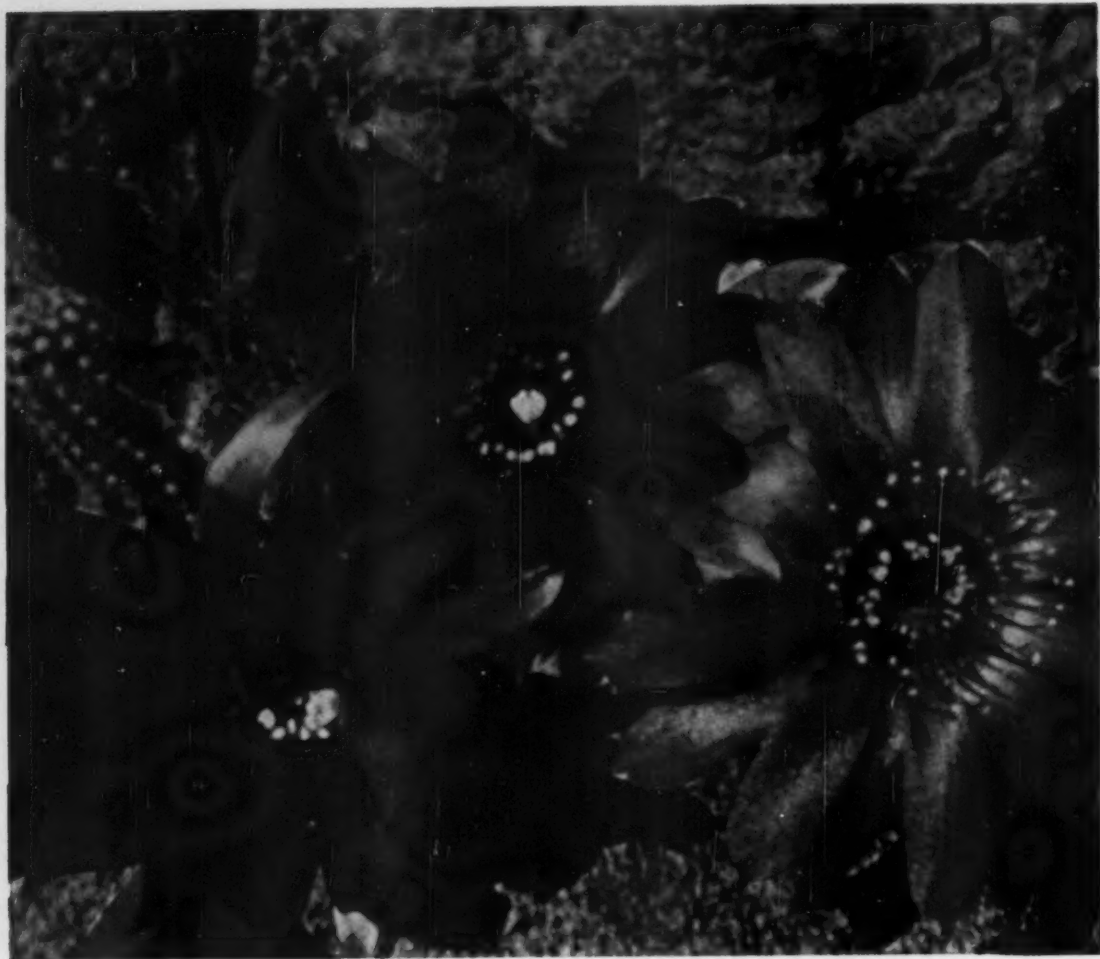
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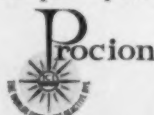
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Examination for the Associateship (A.S.D.C.) 1961

The eighth examination for the Associateship of the Society of Dyers and Colourists was held in May 1961. The question papers and the report of the Examinations Board are set out below. It is hoped that their publication will prove helpful both to prospective candidates and to teachers and will inform members of the Society and others of the standard of knowledge expected from candidates for the Associateship.

Examination Papers

Papers A and B are taken by all candidates; Papers C, D, and E are taken by a candidate only in his chosen branch of tinctorial technology. Further, in Paper E a candidate has a choice of subject—analytical and testing techniques or the organisation of production and economics.

Paper A

General Chemistry and Properties of Dyes, Pigments, Auxiliary Products

General Chemistry and Properties of Organic High Polymers

10 a.m.–1 p.m. on Thursday, 25th May 1961

(SIX questions only to be attempted, three from each section of the paper)

SECTION I

1. What is the importance of the coupling reaction in colour chemistry? Discuss the mechanism of this reaction, giving an indication of the experimental evidence where possible.

2. Give the chemical evidence for the structure of indigo and describe the preparation of indigo from aniline.

3. Give examples of the use of phthalic anhydride in the synthesis of dyes.

4. Compound A ($C_7H_{10}N_3O_3S$), when treated with hydrochloric acid and sodium nitrite, yields a clear solution which, when added to an alkaline solution of acetoacet-p-chloroanilide gives compound B ($C_{17}H_{17}ClN_4O_5S$). B, when heated with cobaltous sulphate in dilute sodium hydroxide solution, yields C ($C_{34}H_{32}Cl_2CoN_8O_{10}S_2$)$^-Na^+$, which is suitable for dyeing wool from neutral dyebaths.
   Write equations showing the formation of C from A, and discuss the application of C to wool.

5. Describe methods for the preparation of triarylmethane dyes.

6. Describe the preparation of one surface-active agent suitable for (a) improving the wet-fastness of dyeings of direct dyes on cellulose, and one for (b) restraining the uptake of leuco-vat dyes. Discuss the mode of action of each agent.

SECTION II

7. Describe the essential features of addition polymerisation. What are the principal differences between addition and condensation polymerisation?

8. Write a short essay on **one** of the following topics—
   (a) Polyamides
   (b) Thermosetting resins
   (c) Naturally occurring 'condensation' polymers.

9. Describe briefly two absolute methods of measuring the molecular weights of polymers. Indicate the limitations of the methods you describe.

10. Show how (a) the glass-transition temperatures of amorphous polymers, and (b) the melting temperatures of crystalline polymers are related to molecular structure.

11. Write brief notes on **three** of the following topics—
   (a) The structure of diene polymers
   (b) Polymer fractionation
   (c) Copolymerisation
   (d) Plasticisation
   (e) Interfacial polycondensation
   (f) Ionic polymerisation.

12. Outline the chemical reactions taking place during the vulcanisation of natural rubber. Describe the alteration in properties brought about by vulcanisation. What are the general requirements necessary for a polymer to exhibit rubber-like elasticity?

Paper B
Theories of Colour
Relation of Colour to Constitution
Colour Assessment and Colour Fastness

2–5 p.m. on Thursday, 25th May 1961

(*FIVE questions only to be attempted*)

1. What are the essential components of a spectrophotometer? What wavelength range would you specify in an instrument for general use in a dyehouse laboratory? Describe briefly how you would check the calibration of a new instrument which you suspected might have been badly shaken in transit from the manufacturers.

2. State Beer's Law and Lambert's Law. How are they used in practical absorptiometry and what precautions need to be taken to ensure that they are valid? Illustrate your answer by a practical example.

3. Discuss briefly the meaning of **four** of the following terms—

(a) Uniform chromaticity diagram
(b) Standard observer
(c) Dichroic colours
(d) Colour temperature
(e) Brightness
(f) Extinction coefficient
(g) Isosbestic point.

4. The loss in depth on washing of a cotton fabric dyed with a mixture of two direct dyes, A and B, is determined by extracting 5-g samples of unwashed and washed fabric with the same amounts of aqueous pyridine. Table I gives the absorption spectrum of each extract together with the absorption characteristics of a 1 g per litre solution of each dye. Calculate the percentage loss of each dye during washing and state what change in the colour of the fabric is suggested by this result.

TABLE I
Optical density

Wavelength (mμ)	Dye A 1 g/l	Dye B 1 g/l	Extract solution Unwashed fabric	Extract solution Washed fabric
400	0·13	0·53	0·29	0·21
425	0·14	0·56	0·31	0·22
450	0·15	0·63	0·34	0·25
475	0·17	0·72	0·39	0·28
500	0·21	0·85	0·47	0·34
525	0·27	0·83	0·49	0·36
550	0·40	0·74	0·54	0·38
575	0·60	0·60	0·60	0·42
600	0·76	0·40	0·62	0·42
625	0·89	0·16	0·60	0·36
650	0·95	0·00	0·57	0·38
675	0·87	0·11	0·57	0·38
700	0·41	0·34	0·38	0·27

5. Describe briefly what is meant by a "colour solid" and explain why the limits of attainable purity depend on surface reflectance. For a blue and a yellow dye sketch the build-up loci on a vertical section through a colour solid. What is the value of such a solid in comparing the ranges of colours attainable with different classes of dyes?

6. Describe the way in which standard grey scales for assessing colour changes during fastness testing are defined and used. A person unfamiliar with the use of these scales attempts to determine fastness to washing by estimating absorptiometrically the percentage loss of dye from a fabric. Discuss the limitations of this approach. Do you consider that the results could be converted to ratings against a grey scale?

7. Describe in detail one well-known colour atlas. Indicate clearly the nature of the colour space on which the atlas is based. Illustrate briefly the value of the atlas in any one branch of industry or the arts.

8. Describe one attempt which has been made to use instrumental techniques for predicting dyeing recipes or paint formulations. What requirements would have to be met before such an approach could equal in accuracy the trained eye of the colourist? What particular advantages would a successful method offer?

9. Discuss the relation of colour to constitution for any one chemical type of dye. How is the colour obtained with a particular dye affected by the substrate?

10. Table II gives the trichromatic co-ordinates for selected monochromatic radiations and also for a painted card A and a magnesium oxide surface in Illuminant C.

TABLE II

Wavelength (mμ)	x	y
380	0·174	0·005
500	0·008	0·538
580	0·513	0·487
700	0·735	0·265
Card A	0·208	0·221
Magnesium oxide	0·310	0·310

The colours of two other painted cards, B and C, in Illuminant C are defined in terms of their dominant wavelengths and excitation purities as follows—

	Dominant wavelength (mμ)	Excitation purity
Card B	580	0·7
Card C	Complementary 500	0·5

What are the trichromatic co-ordinates of cards B and C? If the three cards could be made to form sectors of a disc, which could be spun to fuse the colours, what fraction of the disc would card C have to occupy in order that the resulting colour in Illuminant C should match the magnesium oxide in all but lightness?

11. Write a short essay on **one** of the following topics—

(a) The measurement of whiteness
(b) Colour tolerance specification
(c) Applications of colour theory in printing.

BRANCH 1— TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES
Paper C
Properties of the Materials and Finished Products and Fundamental Mechanisms of the Industrial Processes

10 a.m.–1 p.m. on Friday, 26th May 1961

(*SIX questions only to be attempted*)

1. Write an essay on the mechanisms of dyeing of reactive dyes on cellulose.

2. The term *affinity* has two different meanings, depending on whether it is used by a dyer or by a physical chemist. Explain the difference between the two meanings.

3. Discuss the possibilities and limitations of the application of pigments to textiles.

4. Describe briefly the types of preparation which are available for azoic printing on cellulosic fibres. Discuss, from a colourist's point of view, the advantages and disadvantages of the azoic style, and how these may vary according to the type of preparation employed.

5. How is the activation energy of dyeing derived from times of half-dyeing? What values for the activation energy would you expect for (*a*) direct dyes on cellulose, and (*b*) disperse dyes on Terylene?

6. Discuss the causes and the prevention of "skitteriness" in wool dyeing.

7. Discuss the methods employed for the fixation of disperse dyes printed on polyester fibre fabrics. To what extent does the method of fixation influence choice of dye, and why?

8. Discuss the effect of resin-finishing treatments on the properties of dyeings on cellulosic materials.

9. Compare the dyeing properties of casein fibre with those of wool, and the dyeing properties of cellulose triacetate with those of secondary cellulose acetate. Explain how any differences arise.

10. Why are the following aftertreatments used and how do they work?
(*a*)   Soaping of azoic dyeings
(*b*)   Reduction-clearing of dyed Terylene
(*c*)   Treatment of sulphur dyeings with dichromate and copper sulphate.

11. What is the purpose of steaming printed fabrics? Discuss the steaming of vat dyes printed by the sulphoxylate–potash process, with regard to conditions in the steamer and the reactions taking place in the printed portions of the fabric.

Paper D
Industrial Procedures and Plant
2–5 p.m. on Friday, 26th May 1961
(*SIX questions only to be attempted*)

1. What are the fastness requirements for a warp-knit nylon fabric intended for ladies' underwear? Describe the preparation, dyeing, and finishing of such a fabric.

2. Give a diagram of and discuss recent developments in **one** of the following types of dyeing machine—
(*a*)   hank-dyeing
(*b*)   cone- or cheese-dyeing
(*c*)   loose-stock dyeing.

3. Discuss causes of unlevelness in beam, cone, or cheese dyeing and state what precautions may be taken to overcome them.

4. **Either** Describe a method of bleaching an all-wool hand knitting yarn to give maximum whiteness. Discuss the fastness to light and to washing of the white obtained.
Or Describe a continuous bleaching process for cotton interlock or cellular knit underwear fabric. Give reasons for your choice of bleaching agent.

5. How would you prepare and dye to a solid medium depth **two** of the following—
(*a*)   80/20 Acrilan/wool jersey fabric
(*b*)   50/50 wool/nylon half-hose
(*c*)   50/50 wool/cotton, nylon-spliced half-hose
(*d*)   80/20 Courtelle/wool jumper.

In each case state what standard of fastness you would try to achieve.

6. Write an essay on **one** of the following subjects—
(*a*)   Modern shrink-resist finishes on materials made of or containing wool
(*b*)   Heat-setting of knitted or woven fabrics.

Indicate any effect of these finishes on the dyeing behaviour and fastness properties of the materials.

7. Describe the preparation, dyeing, and finishing of an all-wool taffeta tie cloth.

8. Discuss the function of open-width, multibox washing ranges in modern methods for continuous dyeing. Show how the design of such ranges has developed in order to improve their efficiency in the removal of unwanted material from the fabric.

9. Write notes on the following—
(*a*)   Mélange printing
(*b*)   Moiré finishing
(*c*)   Schreiner calendering
(*d*)   Pad dyeing.

10. What class of dye would you use to produce the following and why?
(*a*)   a navy blue on cotton-drill workman's overall cloth
(*b*)   a cross-dyed red acetate filament warp/ cream cotton weft furnishing fabric
(*c*)   a pale blue on cotton poplin shirting
(*d*)   a beige on Terylene curtain net.
Describe the production of one of them.

11. Describe the following textile drying machines—
(*a*)   Clip stenter
(*b*)   Pin stenter
(*c*)   Cylinder dryer
(*d*)   Air-lay dryer.
In each case give an example in which the use of this machine would be preferred.

12. Give a diagram and describe the operation of **either** (*a*) a roller-printing machine **or** (*b*) an automatic screen-printing machine. What factors must be taken into account when deciding which method of printing to adopt?

Paper E

10 a.m.–1 p.m. on Saturday, 27th May 1961

(**Either** Paper (i) **or** Paper (ii) to be attempted)

(i) Analytical and Testing Techniques

(*FIVE questions only to be attempted*)

Important— *Answer Q.1 and four other questions*

1. Explain the action of a buffer solution. Calculate—

   (a) the pH of a solution containing 0·0034 g-ions of hydrogen per litre

   (b) the hydrogen ion concentration of a solution of pH 6·25

   (c) the quantity of sodium acetate (in g-moles) which must be added to 1 litre of N acetic acid to give a buffer solution of pH 4·5 (K_a of acetic acid = $1·85 \times 10^{-5}$).

2. Describe the methods of paper chromatography and discuss their application to the examination of dyes and fibres.

3. Give details of the tests published by the Society for the determination of the fastness of coloured textiles to—

   (a) perspiration

   (b) dry heat pleating

   (c) chlorinated water in swimming baths.

4. Write a short essay on the use of the microscope in a dyehouse laboratory.

5. Outline the essentials of one method for the quantitative analysis of each of the following fibre blends—

   (a) wool–viscose rayon–secondary cellulose acetate

   (b) wool–Courtelle

   (c) Terylene–cotton

   (d) Rhovyl–nylon.

6. Describe methods suitable for the evaluation of—

   (a) a thickening agent for use in a printing paste

   (b) a fluorescent brightening agent.

7. Describe tests for detecting and estimating damage caused to wool by—

   (a) acid hypochlorite

   (b) alkali.

8. How would you systematically identify the following fibres— Tricel, Acrilan 16, Acrilan, mohair, Terylene, and polyolefin fibre?

9. Discuss the Reports of the Society's Committees on the Dyeing Properties of **two** of the following—

   (a) Direct Cotton Dyes

   (b) Vat Dyes

   (c) Wool Dyes.

10. Describe the examination of dyes to ascertain their suitability for application to goods which are to be given a crease-resist finish.

11. Write notes on the use of **three** of the following in textile analysis—

   (a) ninhydrin

   (b) o-nitrobenzaldehyde

   (c) cuprammonium hydroxide

   (d) carbazole

   (e) titanous chloride.

12. By what tests would you confirm the presence of the following—

   (a) a sulphur dye on cotton

   (b) an azoic dye on viscose rayon

   (c) a 2:1 dye-metal complex on nylon

   (d) a disperse dye on Terylene.

In (b), how would you determine whether the fibre had been dyed or mass-pigmented?

(ii) Organisation of Production and Economics

(*FIVE questions only to be attempted*)

Important— *The terms finishing works and dyeworks cover all firms engaged in the bleaching, dyeing, and printing of textiles.*

1. "The span of control of a manager must be restricted to the number of subordinate functions with which he can have continual contact, and which he can unify effectively". Discuss this statement.

2. What do you understand by functional relationships in industry? Give examples taken from a medium-sized bleach and dyeworks.

3. Outline the purpose and scope of work study.

4. It is said that "piece work is out of date". Discuss this statement.

5. What items are included in the detailed cost per unit of final product in a dyeworks?

6. In textile finishing considerable importance is attached to quality of work. This is taken as an argument for not using standard costing; discuss this.

7. What are the main provisions of the Factories Act as applied to textile finishing works?

8. What do you understand by "labour turnover"? Give some causes and effects of a high labour turnover.

9. How would you regulate the buying and stock control of dyes and chemicals in a medium-sized dyeworks?

10. Write an essay on **one** of the following—

   (a) Fuel economy

   (b) Plant maintenance

   (c) Materials handling.

11. What are the intentions and what may be the economic effects of the Government scheme to reorganise the dyeing and finishing industry in the U.K?

BRANCH 9—PRODUCTION OF DYES, LAKES, AND PIGMENTS

Paper C

Properties of the Materials and Finished Products and Fundamental Mechanisms of the Industrial Processes

10 a.m.–1 p.m. on Friday, 26th May 1961

(*FIVE questions only to be attempted*)

1. Write an essay on **one** of the following topics—

 (a) Dyes containing the triazine ring system

 (b) Anthraquinone vat dyes

 (c) Fluorescent brightening agents

 (d) Inorganic pigments.

Give special attention to the characteristic properties, the chemical constitutions, and the chemical reactions used in the synthesis of members of the group.

2. Discuss *critically* the methods available for the preparation of azopyrazolone dyes and pigments.

3. Outline the chemical reactions employed in the preparation of pigments belonging to any **two** of the following classes—

 (a) Phthalocyanines

 (b) Chrome pigments

 (c) White opaque pigments

 (d) Lake pigments.

Give an account of the important chemical and physical properties of the resulting products.

4. What methods are available for the diazotisation *and* tetrazotisation of aromatic diamines? How have recent physico-chemical studies increased our knowledge of the fundamental processes embodied in these reactions?

5. Outline the chemistry of **two** of the following processes used in the preparation of dye intermediates—

 (a) nitration

 (d) sulphonation

 (c) the Bucherer reaction.

Indicate briefly the application of these intermediates in the manufacture of polyazo dyes and pigments.

6. Outline the chemistry of the manufacture of a green, an orange, and a black vat dye, selecting examples from dyes in current use.

7. Outline the relation between the chemical structure of dyes and related compounds and **two** of the following properties—

 (a) their colour

 (b) their light fastness

 (c) their affinity for the various types of fibres.

8. Give a detailed *critical* account of the method of preparation of **two** triphenylmethane dyes in the green, blue, and violet ranges. (The two dyes chosen must be of different colours.) What is the effect of intramolecular steric hindrance on the properties of dyes of this class?

Paper D

Industrial Procedures and Plant

2–5 p.m. on Friday, 26th May 1961

(*FIVE questions only to be attempted*)

1. Describe the manufacture of one of the following dyes—

(a) Cl—⟨H₃C⟩—N=N—⟨⟩ SO₃Na (commonly known as Lake Red C)

(b) NaO₃S—⟨⟩—N=N—⟨HO⟩ (commonly known as Orange II)

(c) H₃C—⟨CH₃⟩—N=N—⟨HO SO₃Na⟩ SO₃Na (commonly known as Ponceau 2R)
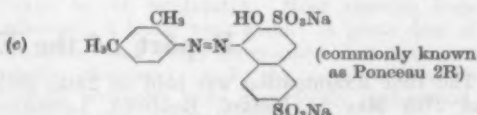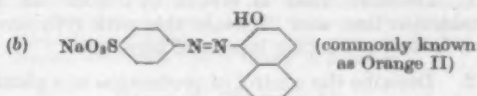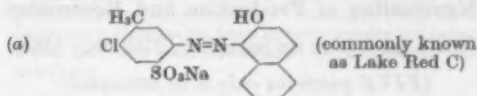
2. Describe in detail both the process and the plant used for the manufacture of a fast-to-light yellow organic pigment. How would you produce it as finely dispersed paste?

3. Describe the manufacture of the yellow solvent dye aminoazotoluene. How may it be converted into a red solvent dye?

4. Describe and sketch an oven suitable for drying lakes and pigments at controlled temperatures.

5. Certain dyes can be converted into phosphomolybdotungstic lakes of increased fastness to light. Give two examples and describe in detail the manufacture of one of them.

6. Describe modern developments in the manufacture of basic dyes.

7. Describe and sketch suitable plant for the manufacture of the dye produced by coupling Tobias acid with β-naphthol. How would you produce a bluish red and a maroon lake from this dye?

8. A batch of an alumina–barium lake of an acid dye is found on drying to be harsh, gritty, and difficult to grind to a fine powder. Give a possible cause of this, and state how you would remedy this trouble in future batches.

9. Discuss the methods employed in the manufacture of any **two** colouring matters which can be used for colouring foodstuffs. What are the chief obstacles which have to be overcome before a new substance can be accepted as a colour for foodstuffs? Give a list of the tests (do not describe the tests)

to which each batch of food dye should be subjected in order that the batch shall meet statutory requirements.

10. What conditions have to be fulfilled in order that a compound produced on a manufacturing scale may be used as a satisfactory pigment for

(a) a printing ink formulation

(b) paint manufacture

(c) a filler in the manufacture of linoleum?

Paper E (ii)

Organisation of Production and Economics

10.0 a.m.–1 p.m. on Saturday, 27th May 1961

(*FIVE questions only to be attempted*)

1. Describe what is meant by "flow" in a production line, and illustrate this with reference to a factory producing lakes and pigments.

2. Describe the control of production in a plant producing azo dyes.

3. What do you consider to be the principal factors governing the scaling up from laboratory to works scale of a process for the production of a dye, lake or pigment?

4. As a manager of a factory producing dyes and pigments you find yourself confronted with a labour shortage. What steps would you take to combat this state of affairs?

5. Discuss the items of cost in the preparation for sale of a dye or pigment.

6. Describe the role of the maintenance engineer in a medium-sized dye factory, and comment on his relation to the production manager.

7. Discuss mechanisation as applied to production of dyes, lakes and pigments.

8. Write an essay on the contribution of the plant supervisor or technician to the economy of production in a lake- or dye-making plant.

9. Describe the methods you would adopt to establish and maintain good labour relations in your plant.

Report of the Examinations Board

The 1961 Examination was held on 25th, 26th and 27th May in Belfast, Bradford, Leicester, London, Manchester, Nottingham, and Paisley. Arrangements were made to hold the examination in Egypt and South Africa but had to be cancelled as the candidates withdrew because of illness and other reasons. It was hoped to hold the examinations in Karachi but the necessary agreement of the Pakistani Government could not be obtained in time.

There were 93 applications to take the examination and 79 candidates actually presented themselves, of whom 51 either took the examination as a whole or papers to complete it, and of these 22 satisfied the examiners. This increase in the number of candidates has not been accompanied by a corresponding increase in the number satisfying the examiners, and there is evidence that many had not prepared themselves adequately. It is unfortunate that many candidates appear to lack fundamental knowledge which is necessary not merely for the purpose of the examinations, but also for the efficient carrying out of their daily work. To illustrate this some of the results of this year's examinations are summarised: 22 candidates took Papers A and B for the first time and 4 passed, 5 took A, B and C similarly and none passed, 31 took the full examination and 8 passed, most of the others failing in A, B and C. Even more disappointing are the results of candidates taking Papers A, B and C for a second time. Of these, 3 out of 9 passed in A, 3 out of 8 in B, and neither of the 2 in C. Candidates were again reluctant to attempt any question requiring calculation. Handwriting was better but many candidates lack ability to express themselves accurately and concisely, and there is still too great a tendency to be satisfied with the recapitulation of factual information without critical discussion. In addition, many candidates are prone to express an opinion without offering any reason for it.

It is interesting to find an increase in the proportion of candidates who chose Paper E (ii) rather than Paper E (i). Many who take Paper E (i) rely on courses of instruction rather than on personal experience.

The candidates called for interview made, in general, a good personal impression. Whatever faults their written work may have had, they were able to express their ideas clearly and thoughtfully by word of mouth. They seemed to be agreeable young people who were often having to bear a good deal of responsibility. They had some quite definite and interesting views on the value and purpose of the examinations and the diploma. There was fairly general agreement that Parts I and II of Paper A would be easier to deal with and fairer to the candidates if they were separate papers, and serious consideration is now being given to this.

Comments on the individual papers follow.

Paper A

62 Candidates sat this paper and 23 passed.

SECTION I

3 Candidates were very good, 9 were good, 11 did not attempt the full number of questions, a number were complete failures. Marks for this part ranged from 0 to 88%.

The examiners' reports may be summarised: there is widespread evidence of a lack, sometimes a complete lack, of preparation. Some candidates did well in only one or two questions in spite of the wide choice. The Society is asked to consider if it could demand evidence of adequate preparation before a candidate is allowed to take the examination.

QUESTION 1 (35)— Poorly answered. There was often insufficient knowledge of reaction mechanisms and confusion of coupling with diazotisation.

QUESTION 2 (24)— 1 Outstanding answer, the rest poor or very poor.

QUESTION 3 (45)— 1 Very good answer, 12 satisfactory. This was better answered than any of the other questions.

QUESTION 4 (19)— A problem question avoided by most; only 4 passed.

QUESTION 5 (26)— Only 4 satisfactory answers.

QUESTION 6 (18)— 1 Very good, 5 good, the rest poor.

SECTION II

A few good papers, the rest disappointing. About half the candidates did not understand the principles of polymer chemistry fully. There were 4 incomplete papers. Many of the answers were disjointed. Marks for this part ranged from 0 to 71%.

QUESTION 7 (53)— 5 Good and 23 satisfactory.

QUESTION 8 (33)— 25 Satisfactory; of these 5 were well-written answers. Undue emphasis was placed on textile applications in 8(a) and 8(b). Only 2 candidates answered 8(c).

QUESTION 9 (30)— 3 Very good answers. Many candidates described measurements of solution viscosity although absolute methods of determining molecular weight were asked for.

QUESTION 10 (8)— All answers satisfactory.

QUESTION 11 (41)— Only 24 satisfactory.

QUESTION 12 (11)— All satisfactory, some were good.

Paper B

General standard poor. 59 Candidates took the paper and only 19 were satisfactory. One candidate was outstanding, and 4 others showed a good grasp of the subject. Questions on absorptiometry were better answered than those on colour physics, which was scarcely understood by many. The question on colour and constitution was well done because triphenylmethane dyes were not excluded from the discussion. There was complete ignorance of published work bearing on questions 5, 8 and 11. Marks ranged from 0 to 63%.

QUESTION 1 (42)— Candidates failed to read the question carefully. Photometers were described but the functions of the essential components were not understood. Few knew what wavelength range would be needed in a dyehouse laboratory. Many omitted a check of the wavelength scale.

QUESTION 2 (53)— Fairly satisfactory. Some candidates could not connect the absorption laws with their applications. Few candidates knew that optical densities in mixtures are additive.

QUESTION 3 (58)— The weakest answers were to sections a, b and c. The meaning of the term "brightness" was understood by few.

QUESTION 4 (43)— A simple question which could have been worked out in 3 or 4 minutes if candidates had based their calculations on densities at wavelengths of both 575 and 650 mμ instead of at one of them only. Candidates also went wrong in not appreciating the simple proportions between weight of dye in the fibre and weight in the extract.

QUESTION 5 (4)— Colour representation appears to be regarded as a two-dimensional concept.

QUESTION 6 (8)— The definition of grey scales was well understood but not their use.

QUESTION 7 (30)— Layout of colour axes was well understood but little reference was made to the system of spacing or to the axes which define a colour space.

QUESTION 8 (9)— One or two candidates had read Atherton's papers but with little understanding.

QUESTION 9 (19)— Basic dyes were the most generally quoted examples. The effect of substrate was imperfectly understood.

QUESTION 10 (9)— A simple question for those who plotted the results graphically. Candidates may have expected a calculation from optical density measurements only and were intimidated by trichromatic co-ordinates.

QUESTION 11 (16)— Only one candidate obtained a complete pass mark. The instrumental measurement of whiteness appeared to be unknown, and colour tolerance specification has not been properly studied. No-one dealt with colour theory in printing.

Paper C (Branch 1)

Taken by 41 candidates. Most answers were satisfactory, 5 being very good. A great deal of information had been memorised but was not critically discussed, and the applications to industrial processes were imperfectly understood. Answers to the printing questions were generally poor and only a few of the candidates who chose them knew clearly what they were writing about. The answers to the dyeing questions were much better. Spread of marks was from 2 to 77%.

QUESTION 1 (22)— Generally satisfactory.

QUESTION 2 (22)— Generally satisfactory.

QUESTION 3 (23)— Only 7 satisfactory answers. Little knowledge was shown of the use of resin-bonded pigments in dyeing and printing.

QUESTION 4 (8)— 2 Very good, 3 satisfactory. Some candidates tended to emphasise the chemistry without making its application clear.

QUESTION 5 (17)— Generally satisfactory.

QUESTION 6 (26)— Generally satisfactory.

QUESTION 7 (9)— 3 Satisfactory answers, the main failure being lack of critical discussion.

QUESTION 8 (29)— Too many candidates gave a good deal of irrelevant information about resin chemistry in general.

QUESTION 9 (19)— Satisfactory; some very good answers.

QUESTION 10 (34)— Many good answers and only two poor ones.

QUESTION 11 (12)— In spite of the popularity of vat printing this was not widely chosen and there were only 1 good and 2 barely satisfactory answers. The functions of steaming were not understood.

Paper D (Branch 1)

39 Candidates took the paper and all but 2 passed. The general standard was reasonably good. The main faults were untidiness, poor presentation, and inadequate sketches. Marks ranged from 33 to 78%.

QUESTION 1 (22)— Over-elaboration was evident in some cases, and methods suggested took into account neither the final properties of the cloth nor the uses for which it was intended.

QUESTION 2 (22)— Descriptions of machines good, sketches poor.

QUESTION 3 (19)— The causes of unlevel dyeing were often not given and methods of overcoming unlevelness were not well understood.

QUESTION 4 (23)— Generally satisfactory.

QUESTION 5 (17)— Reliance was placed on auxiliary products rather than pH and temperature control. There were some good answers.

QUESTION 6 (26)— Many considered it impossible to obtain uniform shrink-resisting properties. The importance of heat-setting was well appreciated. Good descriptions of methods were given.

QUESTION 7 (3)— Two very good answers.

QUESTION 8 (9)— Generally satisfactory.

QUESTION 9 (19)— A fairly straightforward question. Few difficulties were experienced.

QUESTION 10 (34)— Many good answers.

QUESTION 11 (27)— Satisfactorily answered except that diagrams were often poor.

QUESTION 12 (11)— Moderately well answered. Sketches were often poor. Poor arguments were given about the relative merits of screen and roller printing.

Paper E (i) (Branch 1)

17 Candidates, 16 of whom were satisfactory. Some good answers were given, but most were not well written, although descriptions rather than arguments were mostly demanded. Spread of marks was 29 to 76%.

QUESTION 1 (Compulsory)— 2 Candidates did not attempt the question. Several incorrect definitions of pH were given, and there were many incorrect answers, largely because of failure to use logarithms and indices correctly.

QUESTION 2 (6)— Adequate on the practical side. Applications for the examination of fibres were little mentioned.

QUESTION 3 (5)— All but 2 were fairly good.

QUESTION 4 (11)— Few good answers; some candidates had no practical experience of microscopy.

QUESTION 5 (11)— Well answered by most candidates, though few saved time by giving a general introduction to the analysis of blends.

QUESTION 6 (2)— Good answers.

QUESTION 7 (10)— Mostly good, but several candidates were confused about the reactions to a specific test of wool damaged in different ways.

QUESTION 8 (5)— Generally good.

QUESTION 9 (5)— Two candidates gave good discussions of the Reports and not merely details of the tests.

QUESTION 10 (3)— One poor because of lack of experience.

QUESTION 11 (4)— Ninhydrin was not dealt with very fully and part (e) was unanswered.

QUESTION 12 (6)— One poor answer only.

Paper E (ii) (Branch 1)

23 Attempted the paper, 2 were very good and 15 satisfactory. This is the largest number yet to attempt Paper E (ii) and indicates growing interest in these important subjects. When interviewed, some candidates said that they had chosen E (ii) because they thought that their experience of analysis had been sufficiently tested in other examinations. General standard was good but there was some poor spelling and presentation. It should be stressed that in this examination the principles of organisation and economics in the tinctorial industries are asked for and not the technical details of production. The spread of marks was 33 to 80%.

QUESTION 1 (10)— Fairly good answers that showed the principles were understood.

QUESTION 2 (6)— A fairly simple question which some candidates were not very clear about.

QUESTION 3 (21)— Most candidates showed good understanding but some were not clear about the difference between method study and time and motion study.

QUESTION 4 (9)— Some thoughtful answers, but too much personal opinion without any argument.

QUESTION 5 (17)— Although a popular question the answers were not very good. There seems to be great lack of knowledge of elementary costing.

QUESTION 6 (5)— The point of this question, that matters of "finish" and "handle" are not easily definable, was not clearly grasped.

QUESTION 7 (0)— It is significant that no candidate attempted this question.

QUESTION 8 (13)— All the answers were good. It seems to be a problem to which the candidates had given considerable thought.

QUESTION 9 (12)— Another popular question with all those answering it appreciating the need for good stock control, but not many making the point of capital outlay.

QUESTION 10 (18)— Many candidates showed good appreciation of materials handling and the average answer was adequate.

QUESTION 11 (3)— A difficult question. One answer was outstandingly good.

Branch 9

Papers C, D and E (ii) were taken by only one candidate this year.

As a result of the written examinations and interviews, it is recommended that the 22 candidates listed in the Appendix* to this report be elected Associates of the Society.

C. O. CLARK (*Chairman*)

R. L. ELLIOTT	J. V. SUMMERSGILL
W. PENN	H. A. TURNER
C. B. STEVENS	T. VICKERSTAFF

* See pages 440–441.

Proceedings of the Society

The Development of a New Shrink-resist Finish for Wool

A. N. DAVIDSON

It is shown that the treatment of wool materials in a mixed bath containing peracetic acid and sodium hypochlorite gives a high degree of unfeltability together with a soft handle, a good colour, and a slight increase in weight. Laboratory experiments and mill trials are described and some properties of the treated wool materials are indicated.

Introduction

Since the observation by Knecht [1] in 1892 that wool loses its power to felt after treatment in an aqueous solution of chlorine, the reaction between wool and chlorine has received considerable study. Many modifications of the original process have been made and the development of other types of process for the production of shrink-resisting wool has resulted. These methods have included treatment with chlorine gas at reduced [2] and atmospheric [3] pressure; caustic alkalis in non-aqueous solvents [4, 5]; reducing compounds in presence of proteolytic enzymes [6]; oxidising agents (other than halogens), e.g. hydrogen peroxide in presence of traces of metals [7]; potassium permanganate before or after treatment with reducing agents [8], or in presence of sodium hypochlorite [9], or common salt [10]; sodium chlorite [11] followed by chlorine or by papain in sodium bisulphite solution; peracetic [12] and permonosulphuric acids [13]; and resins of various constitutions [14], including the use of resins as a reservoir for chlorine in aqueous chlorine baths [15].

In addition to these chemical treatments, attention has been directed at the Hosiery and Allied Trades Research Association to the influence of the structure of a knitted fabric on its behaviour during laundering. In his work on the shrinkage properties of knitted fabrics, Dutton [16] found that distortion was fairly general in shrink-resisting materials, without the production of felting, and he was able to show that the effect could be minimised by careful attention to the methods used in knitting and finishing. Later work in the same laboratories [17] has shown that the distortion is dependent on the length of yarn (irrespective of nature or count) knitted into an individual loop, and the development of this work has demonstrated that stable fabrics may be knitted by controlling the amount of yarn fed to the needles on the knitting machine. The stable fabrics thus produced are rendered non-felting more easily than others.

After all this work, it might be asked why yet another shrink-resist process is necessary. What are the criteria against which the efficiency of such a process may be judged ? To be wholly satisfactory, a process should—

(a) produce a high degree of unfeltability with the minimum of chemical attack on the fibres,

(b) neither discolour nor alter the handle of the fibres (conversely, the production of a bleached finish or softening of the handle is an added advantage),

(c) give levelness of treatment, including reproducibility of the result,

(d) be suitable for, and applicable to, wool in different forms,

(e) not give rise to any obnoxious smells or attack normal machinery,

(f) have no effect on dyes, and

(g) have a low processing cost and be easily fitted into the normal works' routine.

Judged by these criteria no process at present used is wholly satisfactory.

Although the Perzyme process [6] has given good results under commercial conditions for a considerable number of years, certain defects have been noted. Thus, for fine qualities of wool, treatment for long periods (1–2 h) in a warm (45°C) alkaline (pH 10·5) solution of hydrogen peroxide is needed, and the possibility of excessive attack on the fibres is always present if any mistakes are made. Since the war, a range of powerful oxidising agents (per-acids) has become commercially available. These compounds have been known for a considerable time in the laboratory, but it is only since concentrated (90%) hydrogen peroxide became available that commercial exploitation has been possible. In an examination of the effect of per-acids on amino acids, Toennies and Homiller [18] showed that, of the amino acids present in wool, the per-acids will react only with cystine, methionine, and tryptophan, while Fox [19] suggested that they were without action on the peptide linkage in the time necessary to oxidise the disulphide linkage completely. Rupture of the disulphide linkage appears to be necessary in all shrink-resist finishing of wool by oxidising treatments [20], and thus it seemed likely that the per-acids might be more satisfactory than hydrogen peroxide in a pretreatment in the Perzyme process. Wolsey [12] showed that solutions of peracetic acid will render wool non-felting; however, strong solutions are required and our experiments indicated that the processed wool was badly damaged.

Preliminary Experiments

Experimental treatment of hosiery fabric [21] in acid solutions of per-acids, followed by treatment in either a sodium bisulphite solution containing a proteolytic enzyme (papain) or a solution of chlorine, showed that a reasonable degree of unfeltability could be obtained by the use of a dilute solution of peracetic acid followed by either of the reagents mentioned above. Several works trials of this method using peracetic acid and a sodium bisulphite solution of papain were carried out on yarn, half-hose, and hosiery fabric, in amounts varying from 10 lb of botany yarn (treated in a Hussong machine) to 350 lb of half-hose (treated in a Gorrie side-paddle machine) and very satisfactory results were obtained. After processing, all the treated materials gave very bright level dyeings, although the dye affinity of the fibres was reduced to such an extent that only pale depths could be obtained by normal dyeing methods. This result is similar to that obtained elsewhere [22].

Other per-acids used in the preliminary experiments included performic, permonosulphuric, periodic, and perchloric acids and acid solutions of potassium persulphate, potassium perchlorate, or sodium perborate, while trypsin and pepsin were used instead of papain. None of these other reagents gave a satisfactory non-felting result when commercially practicable quantities were used, except performic and permonosulphuric acids; in these cases, however, poor colour and handle spoiled the result.

Since the use of a two-bath method introduced difficulties under commercial conditions, attention was paid in further experiments to the use of a single-bath method in a mixed solution of a per-acid and sodium hypochlorite. The results obtained from the use of a mixture of peracetic acid and sodium hypochlorite were so satisfactory that in all further work attention was directed to these reagents. The results of a typical experiment in which samples of hosiery fabric (4 × 50 g) were treated in mixed solutions (6 l.) containing different amounts of peracetic acid and sodium hypochlorite, the feltability of the samples being assessed by the standard milling test [23], are given in Table I. It was considered that the results, when judged by feltability, colour, and handle, were sufficiently encouraging for the work to be continued.

Experimental

MATERIALS

Fabric

In all the laboratory experiments, a plain stockinette fabric having 23 courses/in. and made from 2/24s yarn (64s quality wool) was used. After scouring in soap, the fabric was dried at its natural width and with as little length stretch as possible. In works trials, a wide range of hosiery fabrics, half-hose, garments, and blanket fabrics (milled and cellular) has been used, including some mixture fabrics (wool and cotton, wool and rayon). In the treatment of yarns, both botany and cross-bred qualities have been used.

Chemicals

Commercial grades of the following were used— sodium bisulphite (powdered, iron-free), sodium sesquicarbonate, hydrogen peroxide (100 vol.), peracetic acid (36–40%), and sodium hypochlorite (16–18% available chlorine, w/v).

METHODS

Treatment

Experimental treatments of fabrics were carried out either in a bucket under conditions simulating those obtained in a paddle machine or on laboratory winch machines (capacities 6 and 50 l.); yarn in hank was treated on a laboratory Hussong-type dyeing machine (capacity 25 l.). Unless stated otherwise, a liquor : wool ratio of 30 : 1 was used.

Shrinkage Testing

Relaxation— the British Standard method [23] was used.

Washing— a method using an electric domestic washing machine (reciprocating paddle type) was employed under conditions that give equivalent results to those obtained by the British Standard method [23]. The conditions of use were— 50 l. of 0·1% soap solution at 45°C: 1-kg load: 90 min washing time.

Milling— for half-hose and other milling tests the British Standard method [23] was used.

Chemical Tests

The total oxidising power of the solutions was determined by estimating the iodine liberated from an acidified solution of potassium iodide. The per-acids were analysed by the usual method, viz.,

TABLE I

Effect of Concentration of Peracetic Acid and of Sodium Hypochlorite on Shrinkage Resistance in Standard Milling Test

pH	Reagent (% on wt. of wool)		Felting shrinkage (%)		
	Peracetic acid	Chlorine	Length	Width	Area
4·0	2·4	0·5	12·1	4·0	15·6
4·0	2·4	1·0	8·3	0·4	8·7
4·0	2·4	1·5	4·6	−0·9	3·7
4·0	1·2	0·5	14·5	3·1	17·2
4·2	1·2	1·0	6·1	4·8	10·7
4·3	1·2	1·5	5·6	0·4	6·0
4·0	2·4	—	13·2	4·1	16·8
4·0*	—	1·5	8·9	−1·9	7·2
—	—	—	18·2	4·0	21·5

* Adjusted to pH 4 by acetic acid.

estimation of peroxide by titration with potassium permanganate followed by liberation of iodine from an acidified solution of potassium iodide. When necessary, the components of the mixed peracetic acid/sodium hypochlorite solutions were analysed by adding excess of hydrogen peroxide followed by titrating the excess, to give the hypochlorite content, the per-acid then being determined as before.

Results and Discussion

EFFECT OF CONCENTRATION

Further work on the treatment of wool materials in a solution containing peracetic acid and sodium hypochlorite was carried out using a wide range of concentrations of reagents and at different liquor: wool ratios. The results showed that the degree of unfeltability produced depends on the concentration of both reagents, that of sodium hypochlorite having the greater effect. Increasing the concentration of sodium hypochlorite, however, is detrimental to both the colour and handle of the treated material, and it was found that if a ratio of 1 : 2 was maintained between the peracetic acid and the chlorine equivalent of the sodium hypochlorite, the optimum results, as judged by unfeltability, colour, and handle, were obtained. To compensate for changes in liquor:wool ratio, it was found advisable to base the quantities of reagents on the weight of wool; in all subsequent work this method has been followed.

All the samples treated in the mixed bath had a much softer handle and a whiter appearance than those treated in sodium hypochlorite acidified by acetic acid, while, for the botany fabric, the minimum quantity of peracetic acid necessary was 0·75% (on the weight of the wool), mixed with sodium hypochlorite equivalent to 1·5% of chlorine, followed by clearing in sodium bisulphite. Under these conditions, a satisfactory degree of unfeltability was produced with a soft handle and a bleached appearance. When an acid mixture (pH circa 5) has been used, it has not been found necessary to use more than 1% of peracetic acid, and sodium hypochlorite equivalent to 2% of chlorine, even for very fine qualities of wool, to produce a satisfactory degree of unfeltability.

RATE OF REACTION

Analysis of the mixed bath during the treatment of wool materials showed that 50% of the total oxidising power (measured by the liberation of iodine from an acidified solution of potassium iodide) was used in less than 5 min. Laboratory experiments, in which several samples could be introduced into the bath in a matter of seconds, gave no differences between the samples in the degree of unfeltability produced and, on subsequent dyeing, level dyeings were obtained. In the treatment of large batches (200–250 lb) it was not found possible to introduce all the garments in less than 1½–2 min; to determine the effect of this time factor a mixed bath was prepared, and previously wetted-out, numbered samples were added over a period of 2 min and the treatment was continued for 30 min, the samples being agitated by hand-stirring in order to simulate treatment in a side-paddle

machine. After clearing in a cold, acid (pH 4) solution of sodium bisulphite, all the samples were dyed in a single bath to a pastel shade (Coomassie Geranine 2GS (ICI) 0·09%). It was found that the sample introduced first into the mixed bath was dyed to a slightly fuller depth than the others, but in a subsequent washing test no significant differences were found between the felting shrinkages produced, indicating that only a very slight difference in treatment had been given. In further experiments only pale blue or pink dyeings gave this result: samples left white or dyed in deeper colours did not show the difference.

PRESENCE OF SULPHURIC ACID IN PERACETIC ACID

In addition to the rapid initial reaction, another cause of the difference between the first sample and the others was suspected. Commercial peracetic acid contains a small quantity of sulphuric acid and thus a trace of chlorine might be present in the bath. If this were so, it would be expected to react rapidly with the wool first entering the bath. Further samples of fabric were therefore treated, by the method described in the previous paragraph, in baths containing sodium hypochlorite and (a) peracetic acid from which the sulphuric acid had been removed by treatment with baryta, (b) ordinary commercial peracetic acid, and (c) acetic acid equivalent to the total quantity of commercial peracetic acid used in (a) and (b). The results for the rate of decomposition of the baths and the felting shrinkages in the standard washing test are given in Tables II and III.

TABLE II
Loss in Total Oxidising Power (%) during Treatment

Duration of treatment (min)	Treatment with peracetic acid		Treatment with acetic acid
	Baryta added	No baryta added	
2	33	40	72
5	55	65	100
10	65	73	—
15	75	78	—
30	84	86	—

TABLE III
Felting Shrinkage (%) in the Standard Washing Test

	Shrinkage in		
	Length	Width	Area
Peracetic acid: baryta added	3·8	1·3	5·1
Peracetic acid: no baryta added	5·7	−1·3	4·4
Acetic acid	4·8	1·3	6·0
Untreated	21·0	6·4	26·1

The figures given in Table II show that there is a slightly slower reaction between wool and the mixed reagents when baryta is added to the peracetic acid to remove the sulphuric acid, while visual inspection showed that the samples treated in the sulphuric-acid-free mixtures were of a cleaner white than the others. The samples treated in the sodium hypochlorite solution acidified by acetic acid were, as in the earlier experiments, harsher in handle. Addition of samples over a

period of 2 min to a mixed bath containing sulphuric-acid-free peracetic acid and sodium hypochlorite showed that the difference in colour between the first and last samples introduced was not entirely eliminated on subsequent dyeing with Coomassie Geranine 2GS. In all subsequent processing, baryta in the form of a slurry with water has been added to the peracetic acid before admixture with the sodium hypochlorite. The precipitated barium sulphate has no detrimental effect on the treatment.

OCCURRENCE OF YELLOWING

When the method suggested was applied to large batches (200–250 lb) of hosiery fabric, both white and "natural", in the mill, it was found that the fabric became a distinct orange yellow and that during the treatment a smell resembling that of chlorine dioxide was apparent. The original colour of the fabric was restored during the clearing process (with sodium bisulphite) or by sulphur stoving. This result could not be reproduced in the laboratory, and as different scouring methods were used in the mill and in the laboratory, the effect of different scouring techniques on the process was examined. Samples of fabric were scoured in solutions containing the following detergents— soap and soda ash; soap and ammonia; soap; Penetrax (a detergent of the ethylene oxide type); Penetrax and salt; Teepol (s-alkyl sulphates); Teepol and salt. After rinsing and on treatment in the mixed solution of peracetic acid and sodium hypochlorite, it was found that the sample scoured in soap and ammonia gave an identical result to that obtained in the mill, i.e., liberation of chlorine dioxide and production of a golden yellow colour on the wool. It has not been found possible to obtain chlorine dioxide from the mixed reagents by addition of a trace of ammonia: the addition of a trace of ammonia and wool has always given this result. In subsequent processing only materials that have been scoured in an ammonia-free detergent solution have been used.

ADDITION OF REAGENTS

As it had been shown that difficulties might arise in dyeing after the application of the shrink-resist finish, several methods of applying the reagents were tested on fabrics and garments. In both types of finishing, viz., of fabric on a winch machine and of garments on a side- or overhead-paddle machine, the best results were obtained by adding the reagents to the machine while the wool materials were in motion, allowing 2–3 min for mixing after the admission of the first reagent before adding the second, thus assisting a level impregnation. On the winch it was found to be preferable to run the fabric in less than the final volume so that the additions were made while water was still entering the machine. On the paddle machine, the additions were made from the side of the machine, care being taken to ensure that the reagents were not poured directly on to the wool. Differences were noticed in the results according to the order in which the reagents were added. When peracetic acid was added first, chlorine dioxide was produced immediately the

sodium hypochlorite was added, but this reaction stopped as soon as the solution reached pH 5. When the sodium hypochlorite was added first, a reduced level of unfeltability was obtained on the treated garments. Thus, in the treatment of ingrain half-hose (approx. 300 lb in 800 gal of solution), using the same amounts of reagents, the standard milling test on samples taken after processing gave the results shown in Table IV.

TABLE IV
Felting Shrinkage (%) (Length only) of Half-hose in the Standard Milling Test

Method of treatment		Felting shrinkage (%)
Peracetic acid added first	(i)	10·5
	(ii)	10·0
	(iii)	9·0
Sodium hypochlorite added first	(i)	13·2
	(ii)	13·4
	(iii)	14·1

The probable reason for the difference in feltability produced lies in the fact that, when wool is added to an alkaline solution of sodium hypochlorite, the reagent is rapidly absorbed by the wool. The quantity absorbed will not affect the feltability of the wool, but will reduce the effective concentration in the bath for reaction on the addition of the peracetic acid. This result is illustrated by the figures given in Table V, obtained from an experiment in which wool fabric was added to dilute alkaline sodium hypochlorite solutions containing 0·67 g/l. of available chlorine and adjusted to pH 7·5, 8·5, 9·5, and 10·5 by addition of acetic acid. The strength of the sodium hypochlorite solution was determined in the usual way.

TABLE V
Loss of Oxidising Power (%) of Sodium Hypochlorite Solution during Reaction with Wool Fabric

Duration of treatment (min)	pH of solution			
	7·5	8·5	9·5	10·5
2	66	37	23	15
5	92	62	56	28
10	99	87	67	43
20	100	96	87	64

In spite of this difficulty, continued use of the method has shown that satisfactory results are obtained on garments in either a side- or an overhead-paddle machine and on fabric in a winch machine. Later work has shown that a better colour is obtained if a warm (45°C) solution is used in place of a cold solution of sodium bisulphite: this result is in contrast to that obtained in the two-bath method, when warm sodium bisulphite solution gave a yellow colour to the wool.

HANK AND PACKAGE TREATMENT

Treatment of yarn in hank or on cheese raises several problems and presents many difficulties. In either form, a solid package is presented to the treatment liquor, and for a satisfactory result to be obtained all parts of the charge must be treated in a level manner. In dyeing this can be achieved by boiling and the use of levelling agents, but in a

shrink-resist finish that is applied at low temperatures other means must be found. The normal acid reaction is, as has been shown, fairly rapid, making level treatment impossible with a solid package. The fact that an alkaline solution reacts more slowly with wool offered a possible solution.

Knitted fabric was treated in mixed solutions of peracetic acid and sodium hypochlorite at a constant liquor : wool ratio of 30 : 1, the solutions containing different amounts of the reagents at a fixed ratio of 1 part of peracetic acid to sodium hypochlorite equivalent to 2 parts of active chlorine, adjusted to different pH values and at temperatures of 41–70°F (5–20°C). The rate of reaction was measured by determining the total oxidising power; the pH changes were measured on a glass electrode and the feltability of the treated fabrics was assessed by the standard washing test. The collected results for the rate of reaction from numerous experiments, both in the laboratory and under commercial conditions, are given in Fig. 1. The variations in feltability after
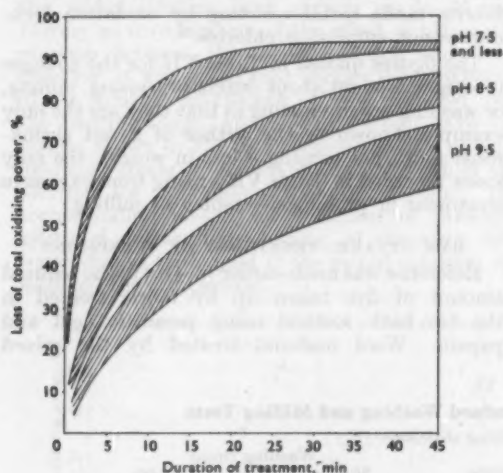

FIG. 1— Rate of loss of total oxidising power at temperatures of 41–70°F (5–20°C)

treatment in a solution containing a fixed amount of reagents at varying pH values, and in a solution containing different amounts of reagents at pH 5·5 and pH 8·5, are shown in Fig. 2 and Fig. 3, respectively.

A study of these results shows that the reaction rate is reduced sufficiently at pH 8·5 to offer the possibility of level treatment under difficult conditions, the time for consumption of half of the oxidising power being 8–12 min under the conditions used. It was also shown that by increasing the liquor : wool ratio from 30 : 1 to 50 : 1 the corresponding time was increased to 15–20 min. Consideration of Fig. 2 indicates that, with fixed amounts of reagents, an acid solution produces a higher degree of unfeltability than does an alkaline solution. The rate of the reaction between wool and the mixed reagents is increased by raising the temperature, and the effect is more pronounced in the slower reactions that obtain under alkaline conditions.
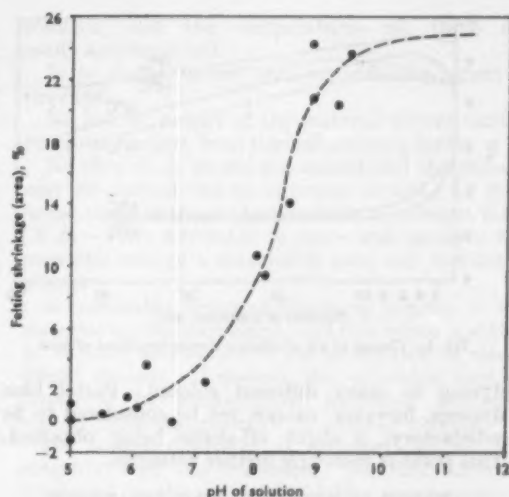

FIG. 2— Felting shrinkage (area) % in a standard washing test after treatment in a solution containing a fixed amount of reagents at varying pH values
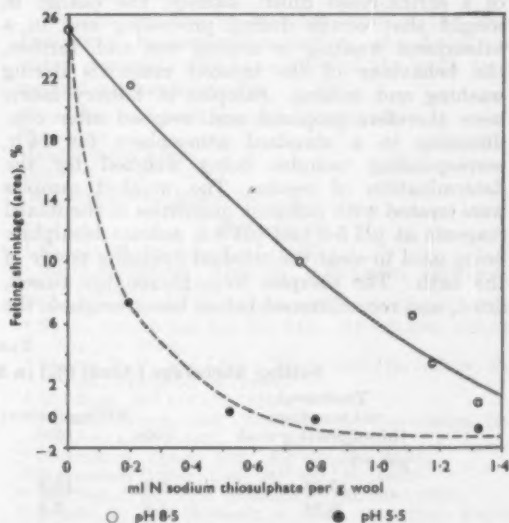

FIG. 3— Variation in felting shrinkage (area) % with degree of treatment

It has been shown that when wool is treated in an alkaline (pH 8·0 to 10·0) sodium hypochlorite solution or an alkaline mixed bath containing peracetic acid and sodium hypochlorite, a rise in the pH occurs during the first 15 min of treatment. A similar rise in pH occurs even if the wool is made acid before being added to the alkaline bath. Treatment of wool in an acid mixed bath gives a continuous, though slight, fall in pH. If the temperature of the alkaline bath is raised, the rise in pH is more quickly counteracted, this probably explaining the quicker reaction rate at higher temperatures. These findings are illustrated by Fig. 4.

The alkaline form of the process has been used on yarn in hank in weights of up to 200 lb with satisfactory results, both in a washing test and in
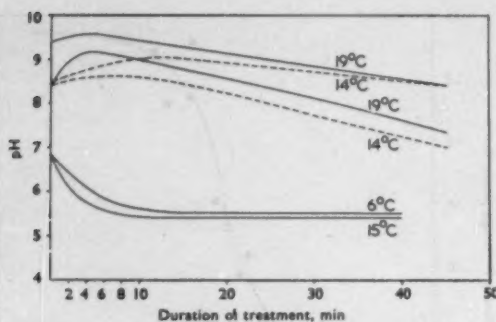
FIG. 4— Change of pH of solution during treatment of wool

dyeing to many different colours. Pastel blue dyeings, however, cannot yet be considered to be satisfactory, a slight off-shade being obtained. This point is receiving further attention.

CHANGE IN WEIGHT DURING TREATMENT, WASHING, AND MILLING

Two other points are of interest in the evaluation of a shrink-resist finish, namely, the change in weight that occurs during processing and in a subsequent washing or milling test and, further, the behaviour of the treated materials during washing and milling. Samples of hosiery fabric were therefore prepared and weighed after conditioning in a standard atmosphere for 24 h, corresponding samples being weighed for the determination of regain. The weighed samples were treated with different quantities of the mixed reagents at pH 5·5 and pH 8·5, sodium bisulphite being used to clear the residual oxidising power of the bath. The samples were thoroughly rinsed, dried, and reconditioned before being weighed, the

regain of each sample being determined. Specimens cut from the treated sample, and from untreated fabric as a control, were used in shrinkage tests (both washing and milling). Each specimen was weighed before and after shrinkage testing, while the corresponding regains were again determined. The results from the shrinkage tests, together with the weight changes, are given in Tables VI and VII. The degree of treatment given to the fabric samples was estimated by the loss of total oxidising power during the processing, which is recorded in ml N sodium thiosulphate per g of wool treated (Column 1 in Tables VI and VII).

The results given in Table VI show that the material treated by the process in either the acid or the alkaline solution is more readily feltable in the milling test than in the washing test. Since prolonged washing failed to increase the felting shrinkage of those samples that gave (initially) a low felting shrinkage in the washing test, it is probable that the method of processing causes the difference. A similar result has been reported for fabrics made shrink-resisting by oxidation techniques not employing chlorine [24].

The figures quoted in Table VII for the changes in weight brought about during processing, milling, or washing are interesting in that they are the only examples known to the author of a wet shrink-resist finish not causing a loss in weight, the only losses recorded in Table VII arising from excessive treatment or prolonged washing or milling.

DYE UPTAKE: TREATMENT OF SHEEPSKINS

Reference was made earlier (p. 410) to the reduced amount of dye taken up by fabric treated in the two-bath method using peracetic acid and papain. Wool material treated by the mixed

TABLE VI

Felting Shrinkage (Area) (%) in Standard Washing and Milling Tests

Treatment ml N sodium thiosulphate/g wool	Felting shrinkage (%)					
	Milling (blows)			Washing (min)		
	1000	1500	2000	30	60	90
pH 5·5						
0·43	7·9	12·3	14·1	−3·1	−0·3	·0·5
0·72	4·2	7·5	7·9	−1·6	0·3	−0·1
0·88	2·0	5·6	7·0	−1·6	−2·4	−3·0
pH 8·5						
0·60	8·6	12·4	15·0	0·5	7·5	10·2
0·92	5·5	9·4	14·1	0·7	2·6	7·9
1·09	6·1	8·0	·9·9	−1·2	−0·4	−0·2
Untreated	8·8	12·8	19·1	8·3	17·6	24·6

TABLE VII

Change in Weight (%) during Processing, Washing, or Milling

Treatment ml N sodium thiosulphate/g wool	Change in weight (%) on dry weight		
	Process	Milling (2000 blows)	Washing (90 min)
pH 5·5			
0·43	1·7	0·2	1·3
0·72	2·0	0·2	1·7
0·88	2·4	−0·2	1·1
pH 8·5			
0·60	0·7	−0·9	0·3
0·92	0·9	−1·3	0·0
1·09	−0·5	−1·5	−1·4
Untreated	—	−0·3	−0·3

solution of peracetic acid and sodium hypochlorite shows a similar result but on a much reduced scale, a noticeable difference in dye uptake occurring only if treated and untreated materials are dyed together. Dyeing tests have indicated that the initial strike of the dye is onto the treated material and that at above 50–55°C the untreated material absorbs more dye. This observation, which is being examined more closely, suggested that the process might be suitable as a pre-dyeing treatment for woolled sheepskins that cannot be dyed at a temperature exceeding 50°C. Several trials, under commercial conditions, have shown that it is possible to dye woolled sheepskins, after processing by the mixed peracetic acid–sodium hypochlorite reagent, to a satisfactory black using Fast Acid Black (CAC) at 45–50°C, the handle of the wool being improved during the treatment.

EFFECT OF pH

In the original experiments it was found that there was only a slight loss in oxidising power of the mixed solution over periods up to 45 min. During an investigation of the effect of pH on the reaction between the mixed solution and wool, however, it was found that greater decomposition of the solution in absence of wool occurred over the neutral range, and a closer study gave the results shown in Fig. 5, in which the loss of total oxidising power is plotted against the pH of the solution for a standing time of 200 min. The corresponding results for solutions of peracetic acid and sodium hypochlorite, of the same concentration as those used in the mixed solution, are included for comparison.
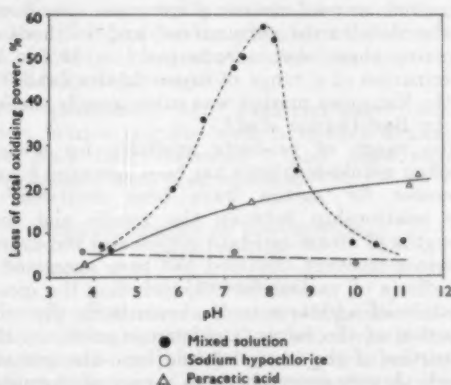

● Mixed solution
○ Sodium hypochlorite
△ Peracetic acid

FIG. 5— Loss of total oxidising power of a mixed standing solution at different pH values, after 200 min (15°C)

In its action on dyed wool, the process gives a similar result to other controlled shrink-resist finishes, the fastness properties of fast dyes being only slightly affected [25, 26].

Conclusions

The main advantages of the new process are—

No special plant is required and all the processing may be carried out in the cold.

The control of the process depends on the chemical analysis of the reagents, the pH of the solution, and the temperature— all these are easily accomplished.

It is cheap to use and no softening agent is required.

No loss in weight of the material occurs during processing, apart from normal scouring losses.

No obnoxious smells are caused and the process may be carried out in ordinary scouring or dye-house sheds without the provision of exhaust fans. (N.B.— It is advisable to store and measure the peracetic acid in a reasonably cool and ventilated place.)

A reasonable degree of bleaching is given to the material by the processing, and this white is stable to washing and assists in the production of clean, bright dyeings. If desired, the whiteness may be improved by conventional bleaching methods, e.g. by hydrogen peroxide or sulphur stoving.

No colour discharge is required, as all the reagents used are colourless, the final clearing treatment in the sodium bisulphite serving merely to reduce any excess of reagents and to brighten the treated material.

* * *

References

[1] Knecht, E., J.S.D.C., 8, 41 (1892).
[2] Wool Industries Research Association (W.I.R.A.), BP 417,719 (1933).
[3] Bradford Dyers' Association Ltd. BP 658,977 (1951).
[4] Tootal Broadhurst Lee Co. Ltd. BP 538,396; 538,428; 538,711 (1939).
[5] Freney, M. R., and Lipson, M., Council Sci. Industr. Res. Aust., Pamphlet 94 (1940).
[6] W.I.R.A. BP 513,919 (1938).
[7] British Cotton and Wool Dyers' Association Ltd. BP 614,966 (1948).
[8] Stevensons (Dyers) Ltd. BP 638,580 (1950).
[9] Stevensons (Dyers) Ltd. BP 569,730 (1945).
[10] McPhee, J. R., Text. Research J., 30, 538 (1960).
[11] Schirlé, C., and Meybeck, J., Bull. Inst. Text. France, No. 45, 31 (1954); No. 46, 29 (1955).
[12] Wolsey Ltd. BP 656,938 (1951).
[13] Stevensons (Dyers) Ltd. BP 716,806 (1954).
[14] Smith, A. R., J.S.D.C., 70, 381 (1954).
[15] Ciba Ltd. BP 675,437 (1952); BP 704,896; 709,377 (1954).
[16] Dutton, W. A., W.I.R.A. Spec. Publ. 9 (1948).
[17] Munden, D. L., J. Textile Inst., 50, T448 (1959).
[18] Toennies, G., and Homiller, R. P., J. Amer. Chem. Soc., 64, 3054 (1942).
[19] Fox, M. R., Ph.D. Thesis, London Univ. (1951).
[20] Speakman, J. B., and Neish, W. J. P., Nature, 155, 45 (1945).
[21] Davidson, A. N., and Preston, R., J. Textile Inst., 47, P685 (1956).
[22] Chesner, L., and Woodford, G. C., J.S.D.C., 74, 531 (1958).
[23] Shrinkage of Knitted Fabrics and Garments containing Wool. BS 1955:1953.
[24] Alexander, P., Symposium on Fibrous Proteins (Bradford: Society of Dyers and Colourists), p. 199 (1946).
[25] Effect of W.I.R.A. Shrink-resist Finish on Wool Dyes (Ciba–Clayton, C.D.C. 67).
[26] Wool Dyes: Effect of W.I.R.A. Shrink-resist Finish on Shade and Wash Fastness (ICI), Technical Information Leaflet Dyehouse No. 536.

A Review of the Chemistry of Thermosetting Resins and Related Compounds and their Application to Textiles

A. R. SMITH

The chemical reactions concerned in the preparation of thermosetting resins based particularly on urea or melamine and formaldehyde are discussed, together with the newer cellulose cross-linking agents, including cyclic urea–formaldehyde derivatives and non-nitrogenous compounds, e.g. glycol acetals and epoxides. The effect of ancillary processes on the properties of resin-treated fabrics and the incorporation of additives in the resin bath are reviewed, with particular reference to the production of smooth-drying cellulosic fabrics, and the problems encountered in producing chlorine-resistant finishes are outlined. Newer developments in the fields of water-repellent treatment and durable flameproofing of cellulosic fabrics are also discussed, and reference is made to the rotproofing of cellulosic materials and the production of bactericidal finishes.

Introduction

Since 1954, when a general review of the application of resins to textiles was prepared by the author [1], some four hundred principal references to the subject have appeared in the literature of Western Europe and the U.S.A. It is probable that a similar number of references to work in other countries, particularly Japan and the Soviet Union, could also be found. Since, however, they appear in journals not readily available in this country, and an accurate assessment of their significance is difficult, they have not been taken into consideration in the present review.

References to some 140 patents and 30 technical papers covering the literature from the original introduction of the crease-resist process by the Tootal Broadhurst Lee Co. in 1926 were assembled in the 1954 paper [1]. The subsequent six years have produced approximately 150 relevant patents and 300 technical papers. The emphasis has thus changed so that, although the number of new patents is considerable, the amount of technical information openly published has increased enormously. Furthermore, the original crease-resist process has been developed to give cellulosic fabrics having "easy-care" or "wash-and-wear" properties, best described, perhaps, as "smooth drying", and a substantial proportion of the literature is concerned with the best methods of achieving this aim. This development has brought with it an increasing interest in finishes for cellulosic fabrics that are resistant to hypochlorite bleaching, and the introduction of "non-chlorine-retentive" finishing agents for this purpose.

Considerable progress has been made in the fields of water-repellent treatment and flameproofing, while other resin applications have received relatively little attention. From the chemist's viewpoint, the products used have become more complex, and mixtures of reagents of different types have been frequently advocated. The mechanism responsible for crease-resistant effects on cellulosic fabrics has been the subject of much speculation, and the theory of cellulose cross-linking, as opposed to the formation of polymeric substances in the amorphous regions of the fibre, has received increasing support, particularly in the U.S.A.

The subject of resin finishing has now become so large that only a few papers of a general nature [2] have appeared in recent years. It is proposed, therefore, in the present review to consider principally the production of smooth-drying cellulosic fabrics, the combination of such effects with chlorine resistance, and the recent developments in water-repellent and flameproofing agents. Other resin applications will be mentioned briefly where literature references are available.

The Crease-resist Process and Smooth-drying Fabrics

The early days of the crease-resist process and its development into a highly successful commercial enterprise were fully described in a recent Mather Lecture [3]. The process was at first applied principally to rayon fabrics and was largely associated with the successful development of spun rayons. The rapid expansion of resin finishing in the post-war years led to the variations in quality of commercial spun-rayon fabrics after creaseproofing, which aroused concern a few years ago. Some of the deficiencies encountered and methods of detecting them were summarised [4] in 1953. An examination of a range of rayon fabrics available on the European market was subsequently carried out by Best-Gordon et al. [5].

The range of products available for crease-proofing cellulosic fabrics has been surveyed [6], and processes for cotton have been reviewed [7, 8]. The relationship between the tensile and tear strengths of crease-resistant cotton and the degree of crease-recovery obtained has been examined [9]; the effects of various factors, including the incorporation of additives in the resin bath, the construction of the fabric, and mercerisation, on the properties of the treated fabric were also investigated. A very comprehensive survey of chemicals and processes used in the production of smooth-drying cotton has recently been made [10].

In 1954 and 1955, the particular advantages of fabrics based on synthetic-polymer fibres were receiving much publicity, one of the main points being rapid drying to a relatively creaseless condition. This led to the use of such phrases as "no-iron", "wash-and-wear", etc., and stimulated the development of cotton fabrics having similar properties.

Smooth-drying finishes for cellulosic fabrics are a simple extension of the principles of crease-proofing developed some thirty years ago, and can be successfully produced by similar methods.

Where, however, the earlier finishes were concerned with recovery from dry creasing, a smooth-drying finish also demands recovery from wet creasing and this generally requires high concentrations of creaseproofing agent. Since the strength of cotton fabrics is reduced progressively as the resin content is increased, this requirement rendered more difficult the achievement of a finished fabric with satisfactory standards of performance. Work on the effects of pre- and post-mercerisation and of additives in the resin bath was stimulated for this reason and comprises a substantial part of the recent published literature. The production of viscose rayon fabrics having high wet crease recovery was even more difficult, owing principally to the greater swelling tendency of wet rayon fibres, and the problems in this case have only recently been solved.

The first developments in the smooth-drying field related to cotton shirtings and emphasised the difficulties of the process. Fabric strength and abrasion resistance were of particular importance and it was found that modifications to the traditional methods of shirt construction were desirable, while tightly woven poplins were not very suitable materials for the smooth-drying process. Furthermore, shirts must withstand severe laundering conditions and the resin finish must therefore be of high durability. A further problem is that in many countries hypochlorite bleaches are employed in the laundering of white fabrics, both commercially and in the home. Urea–formaldehyde and melamine–formaldehyde resins retain chlorine when brought into contact with hypochlorites and, although they behave differently, neither can be considered commercially satisfactory in this connection. This problem has stimulated the introduction of a wide variety of other finishing agents and will be fully discussed later.

The production of "wash-and-wear" white cotton fabrics and the various finishes available have been fully discussed[11], and more recent developments have been reviewed[12-15].
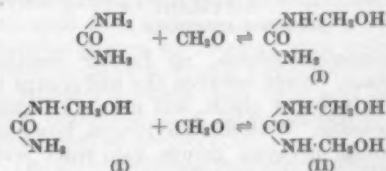
THE CHEMISTRY OF CONVENTIONAL AMINO RESINS
Urea–Formaldehyde

New developments in the use and production of textile finishing agents based on urea–formaldehyde have been relatively few in recent years, but there has been an increased tendency to employ products which have been stabilised by methylation. Methylated urea–formaldehyde precondensates have been shown to possess particular advantages for the production of mechanical finishes on viscose rayon fabrics[16] and in nylon stiffening[17]. They also have the general advantages of stability to storage, increased stability in the impregnating bath, and low content of free formaldehyde.

Little was known before 1950 of the kinetics of the reaction between urea and formaldehyde, but much attention has been aroused in the past ten years. A considerable number of papers has been published[18-20] and the work has been well summarised by Glauert[21].

The primary products of the reaction between urea and formaldehyde are monomethylol urea (I) and dimethylolurea (II)—

$$CO\begin{array}{c}NH_2\\NH_2\end{array} + CH_2O \rightleftharpoons CO\begin{array}{c}NH \cdot CH_2OH\\NH_2\end{array}$$
$$\text{(I)}$$

$$CO\begin{array}{c}NH \cdot CH_2OH\\NH_2\end{array} + CH_2O \rightleftharpoons CO\begin{array}{c}NH \cdot CH_2OH\\NH \cdot CH_2OH\end{array}$$
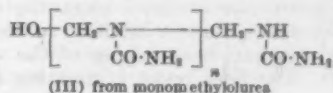$$\text{(I)} \qquad\qquad\qquad\qquad \text{(II)}$$

Both reactions have been shown to be reversible, the forward reactions being bimolecular and the reverse reactions unimolecular. Both are acid–base catalysed reactions, and the rate constant of the first is about three times that of the second. It follows that in aqueous solutions of methylolureas all the above components are present and, although the monomethylol and dimethylol derivatives have been isolated in a reasonably pure condition, an equilibrium mixture is formed when they are redissolved in water. The composition of a solution of monomethylolurea containing 1 mole/l. (9% w/v) is—
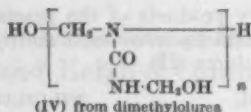
	mole/l.	w/v (%)
Urea	0·26	1·56
Formaldehyde	0·08	0·25
Monomethylolurea	0·56	5·04
Dimethylolurea	0·18	2·15

The treatment of cellulosic fabrics with monomethylolurea and dimethylolurea has been studied by Steele and Giddings[22] but, in view of the behaviour of monomethylolurea in solution, their conclusions about the effect of this compound on the properties of cotton and rayon fabrics may be open to some doubt. Steele and Schiwall[23] have investigated the density changes produced in cellulose by treatment with dimethylolurea, while the reaction of this compound with cotton has recently been examined by Mehta and Mehta[24]. They conclude that the cross-linking of cellulose molecules may not be responsible for the effects obtained, but some of their analytical results have since been criticised by Steele[25]. Some experiments on the acid hydrolysis of dimethylolurea in cotton have been reported[26].
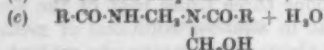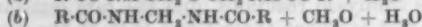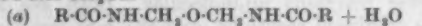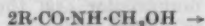
The reactions concerned in the formation of methylolureas are addition reactions, but are followed by condensation reactions which lead to polymeric products. Much of the published information on these products is highly speculative, but it seems generally agreed that the molecular weight of condensates formed in aqueous solution is low (200–500) and that they contain between two and seven urea units per molecule. The polymer at this stage is thought to consist only of linear and branched-chain molecules without appreciable cross linking or network formation, and it is only on further heating in absence of water that the final high polymer is formed. Some possible structures proposed for the intermediate reaction products are—

$$HO \left[\begin{array}{c} CH_2-N \\ | \\ CO \cdot NH_2 \end{array} \right]_n CH_2-NH \\ | \\ CO \cdot NH_2$$

(III) from monomethylolurea

$$\left[HO - CH_2 - N \underset{\substack{| \\ CO \\ | \\ NH \cdot CH_2OH}}{\overset{}{}} H \right]_n$$
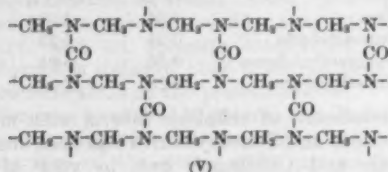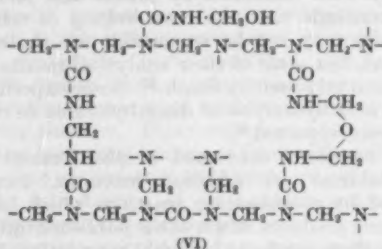
(IV) from dimethylolurea

With monomethylolurea, no further reaction can take place, except between the end-groups to lengthen the polymer chain, and network formation is impossible. With dimethylolurea, however, the remaining methylol groups can react with further –NH– groups or with another methylol group, e.g.—

2R·CO·NH·CH₂OH →

(a)   R·CO·NH·CH₂·O·CH₂·NH·CO·R + H₂O
(b)   R·CO·NH·CH₂·NH·CO·R + CH₂O + H₂O
(c)   R·CO·NH·CH₂·N·CO·R + H₂O
                           |
                           CH₂OH

The products are capable of further reaction, and in theory a completely cross-linked structure containing no –NH– or –CH₂OH groups is possible (V). This theoretical structure is of interest in connection with the problem of chlorine-resistant finishes, which will be discussed later.

–CH₂–N–CH₂–N–CH₂–N–CH₂–N–CH₂–N–
        | | |
        CO   CO   CO
        | | |
–CH₂–N–CH₂–N–CH₂–N–CH₂–N–CH₂–N–
            | |
            CO   CO
            | |
–CH₂–N–CH₂–N–CH₂–N–CH₂–N–CH₂–N–
                                (V)

In practice some formaldehyde is lost during polymerisation by reactions of type (b) above, while the system will become rigidly cross-linked at some intermediate stage and further condensation will be prevented. The final product will therefore contain some –NH– and –CH₂OH groups and could have a more open structure, e.g. VI.

CO·NH·CH₂OH
              |
–CH₂–N–CH₂–N–CH₂–N–CH₂–N–CH₂–N–
        | |
        CO   CO
        | |
        NH   NH–CH₂
        | |
        CH₂   O
        | |
        NH   –N–   –N–   NH–CH₂
        | | | |
        CO   CH₂   CH₂   CO
        | | | |
–CH₂–N–CH₂–N–CO–N–CH₂–N–CH₂–N–
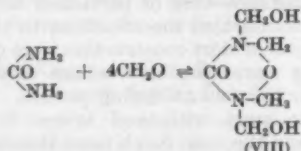   (VI)

Methylolureas are therefore relatively unstable in solution (because of reactions (a), (b), and (c) above) and, where products of very high water tolerance are required, methyl ethers, e.g. VII, are preferred.

NH·CH₂·OMe
           /
CO
           \
NH·CH₂·OMe
           (VII)
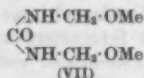
The reactions which take place within the fibres of a cellulosic fabric are not clear. Reactive hydroxyl groups are available along the cellulose chain molecules, so that, in addition to the normal resin condensation, cross-linking of the cellulose is possible. The final result of treating cellulose

with dimethylolurea is probably, therefore, the introduction of some cross-links into the cellulose structure and, at the same time, the formation of polymeric resin aggregates within the amorphous regions of the fibre. Which of these two reactions is responsible for the creaseproof properties imparted to the fabric has not been fully established, but it is clear that such properties can be obtained by other means where resinous polymerisation can be excluded and cross-linking must be responsible. These matters will be more fully discussed later.

The reaction between urea and formaldehyde was thought for many years to be limited to the production of monomethylolurea and dimethylolurea. It has, however, been established by de Jong and de Jonge [18c] that trimethylolurea can exist in solution, and its rate of formation has been determined. Reference has also to be made to compounds formed from one molecule of urea and four molecules of formaldehyde, known as urons (VIII).

CH₂OH
           |
           N–CH₂
NH₂   /   \
  \   /   \
   CO + 4CH₂O ⇌ CO   O
  /   \   /
NH₂   \   /
           N–CH₂
           |
           CH₂OH
           (VIII)

These compounds, the existence of which has not been fully established, would be free from –NH– groups and therefore of some interest in connection with chlorine-resistant finishes. They will be discussed more fully in the relevant section.

Thiourea–Formaldehyde

Resins based on thiourea behave similarly to those based on urea, the initial reaction with formaldehyde taking place under neutral or alkaline conditions. The condensation products were extensively used in the production of moulding compositions in the early days of amino resins and were considered to have a plasticising effect on the moulded materials. The presence of sulphur in the resin molecule resulted in attack on the surface of the steel moulds employed, and these compounds have been replaced in moulding powders by more satisfactory ones. Precondensates based on thiourea are, however, less stable than their urea analogues and rapidly deposit insoluble materials on standing.

Stiffening of nylon dress nets with amino resins is a well-established procedure. It was soon found that such treatments increased the inflammability of the fabric and this increase was associated with the resistance of the resin finish to high temperatures. A skeleton of resin reduces the tendency of nylon to melt on ignition or even prevents melting, and thereby assists propagation of the flame. The effect was particularly noticeable with melamine–formaldehyde resins, which were soon largely replaced by methylated urea–formaldehyde resins [17]. It was shown [27] that addition of thiourea to the resin bath reduced the inflammability of the treated nylon; it has also been proposed to use thiourea–formaldehyde precondensates [28] for this purpose. Unfortunately,

although such precondensates give excellent non-inflammable finishes on nylon, their keeping properties are poor, even after methylation [29]. Modification with various types of amino resin has been suggested, notably urea [30], sulphonated urea [31], melamine [32], and ethyleneurea [33], but a fully satisfactory commercial product has not yet been developed.

Melamine–Formaldehyde

Methylol derivatives are, as with urea, the initial products of the reaction of melamine with formaldehyde but, owing to the greater reactivity of the methylolmelamines, their lower alkyl ethers are principally used in textile applications. For example, the trimethyl ether of trimethylolmelamine, trimethoxymethylmelamine, can be prepared by reaction of formaldehyde with melamine under alkaline conditions to form trimethylolmelamine, followed by methylation using excess methanol under acid conditions. On final polymerisation, the methyl ethers lose methanol and condensation reactions take place which are similar to those which occur with urea–formaldehyde, but more complex. Reaction with more than three molecules of formaldehyde is possible, and hexamethylolmelamine and its hexamethyl ether (IX) have been described. These products contain no –NH– groups and are therefore of interest in the production of chlorine-resistant finishes. It is possible that they are based on the isomelamine structure, since the equilibrium between melamine and isomelamine tends toward the iso compound under the strongly alkaline conditions which favour the formation of hexamethylolmelamine. The hexamethyl ether would then have the structure (X) shown below.

$$N(CH_2OMe)_2$$

$$(MeOCH_2)_2N \quad \text{...} \quad N(CH_2OMe)_2$$

(IX)

(X)
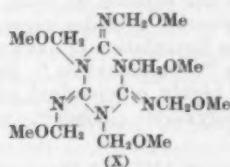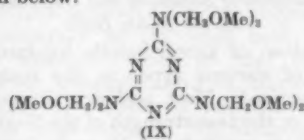
Melamine–formaldehyde resins are more resistant to hydrolysis than urea–formaldehyde resins and have therefore been employed in textile finishing where high durability is required, e.g. in the production of dimensionally stable fabrics or of durable mechanical effects. Their use for shrinkproofing woollen fabrics was at one time the subject of extensive investigation, and for this purpose they were found to give better results when applied as colloidal solutions of relatively high molecular weight. These so-called "acid colloids" are prepared by ageing acid solutions of methylolmelamines or their methyl ethers, and represent an equilibrium between condensation and hydrolysis under the chosen conditions (pH 1·5–3·0). Suitable acids are hydrochloric, formic, acetic, lactic, and glycollic, the organic acids being preferred for textile applications. Their use in shrinkproofing of wool has been described [34] and is the subject of several patents [35], in some of which pretreatment of the wool with hydrogen peroxide is advocated [36]. Another process for treating wool with melamine resins employs a dyeing technique [37] whereby an equilibrium between resin in an acid solution and resin deposited on a fabric immersed therein is established at 80–100°C.

The use of melamine resins for stiffening cellulosic fabrics has also been suggested [38], the acid colloid being again employed.

Miscellaneous Amino–Formaldehyde Derivatives

Apart from urea, thiourea, and melamine resins, the principal products employed in textile finishing are those based on cyclic urea derivatives, which will be discussed in the section on chlorine-resistant finishes. Materials based on the reaction with formaldehyde of other nitrogenous compounds have, however, been proposed from time to time. Among such nitrogenous compounds are dimethyl hydantoin [39] (XI), amides of dibasic acids [40] (XII), acetoguanamine [41] (XIII), and guanylmelamine [42] (XIV). Products based on the reaction of certain aldehydes with ethyleneimine [43], e.g. glyoxal, acrolein, and glutaraldehyde, have also been suggested, while work has been reported [44] on carbonyl bisaziridine (XV), prepared by the reaction of phosgene and ethyleneimine.

(XI)

(XII)

(XIII)

(XIV)

(XV)
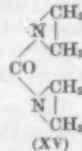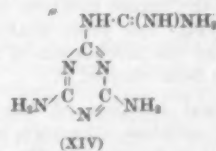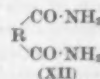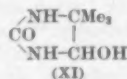
MECHANISM OF THE CREASE-RESIST PROCESS

It was established in the early days of the crease-resist process that the urea–formaldehyde precondensate employed must be of low molecular weight and capable of penetrating the internal structure of the cellulose fibres. It was at first thought that a three-dimensional resin polymer formed within the fibres was responsible for the effects obtained, but more recent work, stimulated by the interest in chlorine-resistant finishes, has suggested that the resin precondensate may react with cellulose to produce cross-links and that these cross-links are actually responsible for the improvement in crease recovery. Evidence in favour of this hypothesis has been advanced by several groups of workers [45–47]. The cross-linking theory has been supported to some extent by evidence obtained from the infrared examination of resin-treated cellulose films, while it is found that crease-resisting properties are imparted to cellulosic fabrics by reagents, e.g. dimethylolethyleneurea, which are incapable of forming a three-dimensional polymer. In contrast, it has been found that other types of polymer, e.g. polyvinyl derivatives,

do not impart crease recovery even when formed within the fibres [48].

The theory of cellulose cross-linking has been recently discussed [49], and changes in the physical properties of cotton produced by cross-linking have been examined [50]. The effect on the solubility of cellulose in cuprammonium solutions produced by cross-linking has been reported [51]. A recent paper [52] which discusses cellulose cross-linking reactions more generally is also of interest in this connection.

The two sides of this controversy have been well summarised by Marsh [53], but no final conclusion seems yet to have been reached. It is almost certain that both cross-linking of cellulose and polymer formation within the fibres take place when cellulosic fabrics are treated with conventional amino resins. Recent work by O'Brien and van Loo [54] suggests that both intermolecular and intra-molecular bonding occur in resin-treated cellulose and that only a relatively small proportion of the resin is actually involved in cross-linking. As melamine resins, which are the most likely to form complex networks, are less efficient than urea resins as creaseproofing agents, it is possible that polymer formation is, in fact, an undesirable side-reaction, but a firm conclusion must await more extensive investigations. Further discussions of cotton cross-linking [55-57] and some microscopical work [58] are also of considerable interest.

The physical mechanism responsible for the production of smooth-drying or "wash-and-wear" cotton fabrics has been discussed [59], and the physical properties imparted to viscose rayon have been recently studied [60]. The relative significance of wet and dry crease recovery in conjunction with smooth-drying has been examined in a recent paper [61], and the mechanism of resin deposition in fabrics has been studied [62].

The actual location of the resinous product within the cellulose structure was established by dye-staining techniques in the early days of resin finishing, as recently described [63]. Best-Gordon [4] has outlined the technique of staining resin-treated rayon fabrics with azoic dyes to show the overall distribution of the resin in the fabric, and has demonstrated the migration of resin towards heated surfaces which can occur during resination. A more refined technique for the location of resin, based on the use of resin precondensates containing radioactive materials, has been successfully exploited by several workers [64-66], of whom Park employed tritium instead of the ^{14}C used by earlier workers. The use of the electron microscope in the location of reaction areas in chemically treated cellulose has been described [67]. Chemical analysis of fabrics treated with amino resins and related products has received attention; methods described in the literature are often of doubtful value when the investigator is examining a fabric of unknown origin, and a combination of a number of test methods with adequate experience in their interpretation is required if reliable results are to be obtained. A useful scheme of analysis has been proposed [68] and a summary of analytical methods from various sources has been published [69].

VARIABLES INVOLVED IN THE RESIN TREATMENT OF CELLULOSIC FABRICS

Fabric Construction

Many resin finishers work on a commission basis and are therefore unable to influence the choice of a fabric for resin treatment. It has clearly been established, however, that the construction of the fabric and the nature of the yarn employed have a profound influence on the results obtained, particularly in regard to the relation between crease-recovery obtained and the strength of the treated fabric. The application of amino resins to yarn has recently been investigated [70, 71], and the effect of fabric construction has received particular attention [72-74].

Effect of Mercerisation and Allied Processes

Mercerisation of cotton or linen before resin treatment was proposed in the early days of resin finishing by the Tootal Broadhurst Lee Co. Since the process has the effect of reducing the crystallinity of fibres based on natural cellulose, it is likely to improve the accessibility to resin finishes and this is reflected in practice by improved fabric strength at a given level of crease recovery. More recent work [75, 76] has confirmed the advantages of this procedure. Pretreatment of viscose rayon fabrics with caustic soda or other swelling agents has recently been proposed for the production of smooth-drying finishes [77]. In this case, the process has the effect of improving the wet crease recovery of the treated material.

Mercerisation after resination has also been proposed and shows considerable advantages [78, 79].

Incorporation of Softeners and Other Additives in the Resin Bath

Modification of fabric handle by incorporating additives of various types in the resin bath is widely practised. Most additives improve either the tensile or the tear strength of the treated fabric, both of which are reduced by resin finishing, and the patent literature includes suggestions for the addition to the formulation of natural or synthetic rubber latices [80], polyethylene [81] (both proposed before 1940), highly polymerised amino resins [82], silicones [83], polyvinyl alcohol [84], and polyvinyl acetate [85].

It has been established [9] that compounds, e.g. polyvinyl alcohol, which stiffen the fabric, improve the tensile strength and reduce the tear strength still further, while softeners have the opposite effect. The only cases in which both tensile and tear strength are improved are those in which elastomers, e.g. rubber latex, are incorporated in the fabric; such additives may produce an undesirable rubbery handle if used in excess. Many are also liable to produce discoloration at baking temperatures or on subsequent exposure of the fabric to light. The addition of butadiene–styrene or butadiene–acrylonitrile latices in this connection has been discussed [86-88], the use of acrylate emulsions has been investigated [89] and the incorporation of styrene–ethyl acrylate copolymers has been reported [45].

Although the use of polyethylene as an additive to resin formulations was patented more than twenty years ago, it is only recently that suitable emulsions have been commercially available; their application has been discussed [90]. The use of thermoplastic additives is open to the objection that the tendency of the treated fabric to wet-soiling is increased. It would appear that the thermoplastic material, which must lie on the surface of the fibres, is softened on immersion in hot washing liquors, and suspended soil can be picked up by the fabric under these conditions. An examination of the problem has been reported [91].

The effect of textile softeners of various types has been fully investigated by the Piedmont Section of the A.A.T.C.C.[92], who have shown that the tear strength and abrasion resistance of the fabric can be improved. Durable water-repellent agents can also be used in reduced quantities as softeners, and those based on stearamide have been frequently employed in this way. The stearamide-modified melamine resins, which contribute to the crease recovery of the treated fabric in addition to improving tear strength and abrasion resistance, are particularly useful. A further development, described by Simpson [93], is the use of silicone softeners, which are similar to silicone water-repellent agents, but may be applied without a silicone catalyst if water-repellency is not required. A recent paper by Marsh [94] reviews the entire subject of the inclusion of additives in the resin bath.

Catalysts

The catalyst or accelerator used to expedite the final polymerisation of the resin in the fabric is a very important factor in the process. Organic acids were first employed for this purpose, but the storage properties of the resin bath were very adversely affected, since at the time urea–formaldehyde precondensates of limited stability were employed. Within a comparatively short time these were replaced by ammonium salts, particularly ammonium dihydrogen phosphate, with considerable improvement in bath stability. During the past few years, the patent literature has contained references to a wide variety of catalysts for which improved bath stability and greater efficiency have been claimed. Among those of some interest are co-ordination complexes of urea with metallic salts such as aluminium sulphate [95], alkanolamine hydrochlorides [96], esters of *p*-alkylbenzene sulphonic acids [97], mixtures of calcium chloride and boric acid [98], hydrogen peroxide [99] (which presumably oxidises formaldehyde to formic acid), ammonium salts of organic sulphonic or phosphonic acids of high molecular weight [100], derivatives of cyanuric chloride related chemically to the reactive dyes [101], and salts such as magnesium dihydrogen phosphate [102], which develop acidity as the solutions become concentrated in the fabric.

A recent development is the use of water-soluble alkyd or polyester resins [103] based on, e.g. polyethylene glycol and succinic acid. These products are of relatively high molecular weight and act as modifiers of fabric handle, but also, owing to their acidity, they can replace conventional catalysts.

In spite of the wide range of products proposed, the principal commercial interest in recent years has been directed towards simple metallic salts, particularly magnesium chloride and zinc nitrate. These substances are capable of developing acidity in the fabric at the temperatures employed in the final baking process but, at the same time, the resin bath remains substantially neutral. Magnesium chloride has proved satisfactory in conjunction with the newer cyclic urea derivatives used in the production of chlorine-resistant finishes, but shows low efficiency when employed with urea–formaldehyde. Zinc nitrate, on the other hand, is extremely effective with both urea– and melamine–formaldehyde resins, as well as with the cyclic urea derivatives. A mixture of magnesium and aluminium chlorides has recently been proposed as a catalyst for the new. non-nitrogenous glycol acetal cross-linking agents [104], but this is relatively ineffective when used with amino resins.

The general subject of catalysis has been reviewed [105, 106], while an elaborate investigation of the relative efficiencies of ammonium salts, alkanolamine hydrochlorides, and magnesium chloride has been carried out [107]. The chemical mechanism of catalysis has recently been examined [108], and the effect of catalysts on the physical and chemical properties of resin-treated cottons has been investigated [109] by microscopical and infrared methods.

Curing Conditions

After application of the finishing formulation to the fabric and subsequent drying, a final curing or baking process is necessary. This is carried out in the conventional process by passing the fabric through a baking chamber. Temperatures of 140–180°C are commonly employed, the baking time being proportionately shorter at higher temperatures. Modern finishing agents tend to require higher baking temperatures for maximum efficiency, and this is particularly so with chlorine-resistant finishes, since incomplete curing can lead to subsequent partial hydrolysis of the finish on storage and development of chlorine-retentive groups on subsequent washing. This will be more fully discussed in the section on chlorine retention.

Severe curing conditions tend to affect the fabric strength adversely, and it has been suggested that curing in presence of steam will produce improved results [110]. It is possible, however, that since water is one of the products of the condensation reaction, this procedure improves fabric strength by reducing the degree of cure. The conditions inside the baking chamber, where both steam and gaseous formaldehyde are present, will vary according to the type of plant used and probably at different stages in the processing of a batch of fabric. Some of these conditions have been investigated [111], but many of the factors involved would merit further examination. The general problem of resin curing has been discussed by Foster [112].

ASSESSMENT OF THE TREATED FABRIC

In the early days of the crease-resist process, methods for measuring dry crease recovery were developed by the Tootal Broadhurst Lee Co. and

have served satisfactorily for many years. However, the introduction of smooth-drying or "wash-and-wear" fabrics has stimulated the development of further tests, and the expanding application of the process has resulted in a considerable increase in the quantity of material to be tested. The Shirley Institute and Monsanto testers appeared to be satisfactory for the measurement of dry crease recovery, but these instruments have been said to assist in the recovery of the creased test specimen; gravitational forces help recovery from creasing in these cases, while opposing recovery when the TBL method is used. For this reason, conversion of results from one system to the other by mathematical calculation is inadvisable. Newer testing devices, e.g. the Stoll–Celanese tester, described by Kaswell [113], the Optical Wrinklemeter [114], and the Electronic Smoothness Evaluator [115] have been proposed, and the automatic measurement of crease recovery by electronic means has been described [116]. It has since been realised that both wet and dry crease recovery are involved in smooth-drying; this problem has recently been discussed [117]. The recovery of the fabric from wet creasing during the drying process is also significant, and an optical method of measuring this has been proposed [118].

In the U.S.A., assessment of the smooth-drying properties of the fabric by visual means has been preferred, and a series of standard fabrics for comparison with the test specimen under standard illumination has been devised [119]. The examination of fabric surfaces under low-angle illumination has also been suggested [120]. An attempt to provide more reproducible standards has been made [121], the use being proposed of plastic replicas of the surfaces of a series of fabrics with a range of smooth-drying properties. It was pointed out, however, that coloured or patterned fabrics should not be compared with white standards, as colour and design influence the judgment of the observer and can mask the deficiencies of the test specimen. Another method of preparing plastic replicas of fabric surfaces has been developed in the U.K. by Sudnik [122], who has recently [123] commented on the relation between his standards and those of Williams [121]. Subjective and objective methods of "wash-and-wear" assessment have recently been compared [124].

One of the problems associated with crease-proof finishes on cellulosic fabrics has been the reduction in abrasion resistance of the fabric following resin treatment. It was mentioned earlier that addition of such materials as softeners and synthetic latices to the resin bath can improve resistance to abrasion, and their efficacy has usually been assessed by means of accelerated abrasion tests. It has been suggested that such tests are unrealistic; nevertheless, they are of considerable value in examining the effect of modifying the fabric finish in various ways. A well-established device for the measurement of resistance to abrasion is the Tootal Ring-Wear machine, but a number of other methods of test have also been employed, including the Stoll Abrasion Tester in the U.S.A. and the Martindale machine in the U.K. An interesting abrasion tester has also been

developed by the Linen Industry Research Association [125]. In the U.S.A., much interest has been shown in the Accelerotor, described by Stiegler et al. [126] and investigated further by Cooke [127]. This machine is claimed to be particularly useful for examining the effects of abrasion at fabric edges, for which the earlier methods of assessment are unsuitable. A modification of the machine to operate under constant power input has recently been described [128]. In the U.K., the Bocking BFT machine [129] has been developed specifically for the examination of resin-treated viscose rayon fabrics. Two separate components of abrasion resistance, flex abrasion and ball abrasion, are determined; these two components are combined into one figure, the Duty Factor of the fabric, by calculating their harmonic mean, and this has been shown to correlate well with the performance of rayon fabrics in actual wear. Results on a variety of commercial fabrics are given in a subsequent paper [130].

Other problems concerned with resin-treated fabrics have also received attention, in particular their resistance to chlorine bleaches, which will be discussed later. The evolution of formaldehyde from resin-treated fabrics on storage has been examined [131, 132], and investigations have been made of the effect of actual storage [133] and of an accelerated storage test [134] on the smooth-drying properties of the treated material.

### Production of Chlorine-resistant Finishes

Although textile finishes based on urea–formaldehyde or melamine–formaldehyde resins have been produced commercially for many years, it is only recently that the problem of retention of chlorine by such finishes when the fabric is treated with hypochlorite bleaches has assumed any importance. In the earlier years, resin finishes were principally applied to rayon fabrics, which are not usually subjected to severe washing conditions, while most of these fabrics were coloured and were therefore unlikely to be treated with hypochlorite. Interest in chlorine retention resulted mainly from the application of resin finishes to white cotton shirts to impart smooth-drying properties, which is a relatively new development.

Investigations were made some years ago of the adverse effects of retained chlorine and their assessment [135], and the problem was examined in relation to commercial resin-treated fabrics [136]. A standard test for damage due to retained chlorine was developed by the A.A.T.C.C. in 1953 [137] and revised in 1959 [138]. Swiss test methods were recently discussed [139].

PRINCIPLES AND MECHANISM OF CHLORINE RETENTION

Whether resin polymerisation or cellulose cross-linking is responsible for the effects produced by applying amino-resin precondensates to cellulosic fabrics, a conventional finish based on urea–formaldehyde or melamine–formaldehyde will contain –NH– groups. When the treated fabric is subsequently exposed to the action of a hypochlorite, chlorine is absorbed with formation of a chloramine—

$$-NH- + HOCl \rightarrow -NCl- + H_2O$$
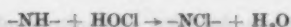
When the chlorine-treated fabric is ironed, hydrochloric acid and nascent oxygen are formed, and chemical damage to the cellulose occurs if the finish is based on urea–formaldehyde—
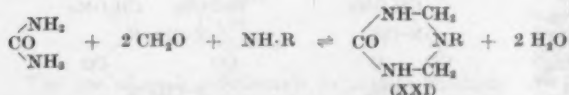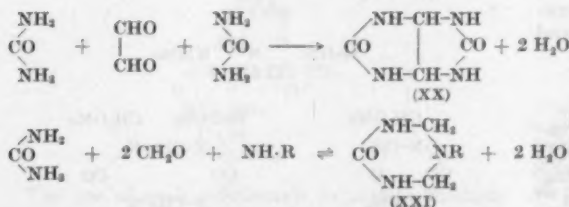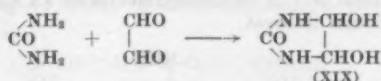
$$-NCl- + H_2O \rightarrow -NH- + HCl + O$$

With melamine–formaldehyde, on the other hand, a chloramine is formed as before, but causes yellowing of the fabric, which does not occur with urea–formaldehyde. No hydrochloric acid is produced on ironing, however, as the chloramine is more stable under such conditions.

One solution to the problem of producing chlorine-resistant finishes is to employ the dimethylol derivative of a substituted urea containing no amino groups; compounds of formulae XVI and XVII have been investigated. According to the literature [140], simple monoalkyl and dialkylureas do not form dimethylol derivatives, so that compounds of formula XVII have been the ones principally examined.

$$CO \begin{matrix} \nearrow NHR \\ \searrow NHR \end{matrix} \qquad CO \begin{matrix} \nearrow NH \\ \mid \quad R \\ \searrow NH \end{matrix}$$

(XVI)   (XVII)

The principal developments in this field have been based on cyclic urea derivatives, e.g. *NN'*-ethyleneurea (XVIII), dihydroxy-*NN'*-ethyleneurea or glyoxal ureine (XIX), and the various triazones or triazinones (XXI).

$$CO \begin{matrix} \nearrow NH_2 \\ \searrow NH_2 \end{matrix} + \begin{matrix} NH_2-CH_2 \\ \mid \\ NH_2-CH_2 \end{matrix} \longrightarrow CO \begin{matrix} \nearrow NH-CH_2 \\ \searrow NH-CH_2 \end{matrix} + 2\,NH_3$$

(XVIII)

$$CO \begin{matrix} \nearrow NH_2 \\ \searrow NH_2 \end{matrix} + \begin{matrix} CHO \\ \mid \\ CHO \end{matrix} \longrightarrow CO \begin{matrix} \nearrow NH-CHOH \\ \searrow NH-CHOH \end{matrix}$$

(XIX)

$$CO \begin{matrix} NH_2 \\ \\ NH_2 \end{matrix} + \begin{matrix} CHO \\ \mid \\ CHO \end{matrix} + CO \begin{matrix} NH_2 \\ \\ NH_2 \end{matrix} \longrightarrow CO \begin{matrix} NH-CH-NH \\ \mid \quad\quad CO \\ NH-CH-NH \end{matrix} + 2\,H_2O$$

(XX)

$$CO \begin{matrix} \nearrow NH_2 \\ \searrow NH_2 \end{matrix} + 2\,CH_2O + NH\cdot R \rightleftharpoons CO \begin{matrix} \nearrow NH-CH_2 \\ \quad\quad NR \\ \searrow NH-CH_2 \end{matrix} + 2\,H_2O$$

(XXI)

(R = Me, Et or C_2H_4OH)

These three classes of compound— ethyleneurea, dihydroxyethyleneurea, and the triazones— are the ones of main commercial importance. Acetylene diureide (XX) will form a tetramethylol derivative in solution which would contain no imino (NH) groups, but this is unstable and loses formaldehyde on being applied to the fabric and subsequently baked, so that the finish becomes chlorine-retentive. The urons would be of obvious economic interest since they are prepared simply from urea and formaldehyde, but again their stability is in some doubt. These points will be further discussed later.

The mechanism of chlorine-retention has been discussed by Howard [141] in relation to dimethylolethyleneurea (DMEU), and more recently by Rumens [142]. The effect of different catalysts and curing conditions has been investigated by Smith *et al.*[143], who showed that better results were obtained with DMEU by hard curing, particularly with metal-salt catalysts. The initial chlorine resistance of finishes based on DMEU is usually satisfactory, but the development of chlorine-retention after washing is a serious defect which has been the subject of considerable investigation. Some of the problems have been discussed [144], while the effect of home washing [145], and the consequences of washing under commercial laundering conditions [146], which in the U.S.A. frequently involve an acid souring process, have been investigated.
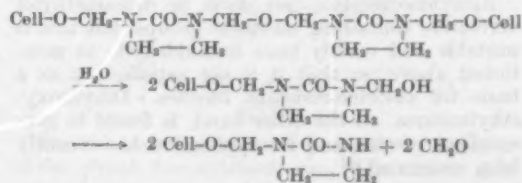
A further problem posed by finishes based on cyclic ureas is the change which takes place on storing the treated fabric [147, 148]. The finish hydrolyses on storage, particularly if the catalyst has not been removed from the fabric by washing-off after resin treatment. This does not result in the immediate development of chlorine-retention, if the original curing conditions were adequate, but serious chlorine damage can be observed on subsequent washing. The mechanism responsible for this behaviour is not completely established, but it is probable that storage, particularly under conditions of high humidity, causes the liberation of free methylol groups—

$$-N\cdot CH_2OR + H_2O \rightarrow -N\cdot CH_2OH + R\cdot OH$$

Subsequent washing results in loss of formaldehyde and formation of –NH– groups—

$$-N\cdot CH_2OH \rightarrow -NH- + CH_2O$$

It has been suggested [149] that breakdown of the ring structure of the ethyleneurea takes place under severe conditions, e.g. commercial laundering. This theory has found support [147], but it does not explain the two-stage process involved in storage followed by washing, or the fact that even a relatively mild wash is sometimes sufficient to develop chlorine-retention after storage. Breakdown of the stable ethyleneurea ring structure is unlikely, and it has been suggested [148] that this two-stage deterioration is more probably due to the presence initially of longer cross-links involving two or more molecules of DMEU—

$$Cell-O-CH_2-N-CO-N-CH_2-O-CH_2-N-CO-N-CH_2-O-Cell$$
$$\quad\quad\quad\quad\mid\quad\quad\quad\mid \quad\quad\quad\quad\quad\quad\mid\quad\quad\quad\mid$$
$$\quad\quad\quad\quad CH_2---CH_2 \quad\quad\quad\quad\quad CH_2---CH_2$$

$$\xrightarrow{H_2O}\quad 2\,Cell-O-CH_2-N-CO-N-CH_2OH$$
$$\quad\quad\quad\quad\quad\quad\quad\quad\mid\quad\quad\quad\mid$$
$$\quad\quad\quad\quad\quad\quad\quad\quad CH_2---CH_2$$

$$\longrightarrow\quad 2\,Cell-O-CH_2-N-CO-NH + 2\,CH_2O$$
$$\quad\quad\quad\quad\quad\quad\quad\quad\mid\quad\quad\quad\mid$$
$$\quad\quad\quad\quad\quad\quad\quad\quad CH_2---CH_2$$

This mechanism is in accordance with the evidence [149] that the average length of cross-links based on DMEU is 1·5 molecular units, and it has been supported [150] in a recent discussion of the problem. A useful series of references to the literature is to be found in another recent examination of the problem [151].

THE CHEMISTRY OF CYCLIC UREA
DERIVATIVES
Ethyleneurea

Various methods for the preparation of ethylene-urea (XVIII) have been proposed, based on the reaction of urea, carbon dioxide, or ammonium carbonate with ethylene oxide, ethylene glycol, or ethylenediamine. The best yield is obtained by reaction of urea with ethylenediamine [152].

The use of dimethylol ethyleneurea in textile finishing for creaseproofing and for the production of durable mechanical effects was patented [153] as early as 1948, before the interest in chlorine-resistance arose. The behaviour of DMEU was compared with that of melamine–formaldehyde by Stam et al.[154], while its effectiveness in cross-linking polyhydroxy compounds was demonstrated by Walter et al.[155], who showed that poly-vinyl alcohol could be insolubilised in this way. The general properties of DMEU and finishes based on it have been discussed in two papers by Marsh [156].

The deficiencies as regards storage and washing of finishes based on DMEU have led to suggestions that it should be used in conjunction with other finishing agents, conventional melamine resins being particularly advocated [157]. Its use in conjunction with certain non-nitrogenous cross-linking agents for cellulose, which will be discussed later, has also been proposed, including its use with acetals [158] and its addition to finishes based on epoxy derivatives [159]. A compound of particular interest is hexamethoxymethylmelamine (IX), which also contains no imino groups and therefore forms a possible basis for chlorine-resistant finishes. Its use in conjunction with DMEU has been suggested [160], and the application of a modified triazine derivative has been proposed [161]. The use of propyleneurea and hydroxypropyleneurea, which are prepared in a similar way to ethylene-urea, has recently been examined [162] and discussed [163].

Dihydroxyethyleneurea and Acetylenediureide

The use of glyoxal as an additive to urea–formaldehyde finishes was proposed at a relatively early stage in the development of resin finishing, but the production of dihydroxyethyleneurea [164] (XIX) or acetylenediureide [165] (XX) by previous reaction of urea and glyoxal is a later development.

Acetylenediureide can form a tetramethylol derivative containing no imino groups, but this is unstable and readily loses formaldehyde as mentioned above, so that it is not satisfactory as a basis for chlorine-resistant finishes. Dihydroxy-ethyleneurea, on the other hand, is found to give excellent results, and its application has recently been examined [166].

The Triazones and Triazines

The reaction of urea, formaldehyde, and a primary amine was investigated some years ago by Paquin [167], who showed that triazinones or tri-azones (XXI) were formed.

Recent work has suggested that these compounds, known commercially as triazones, form a valuable basis for the production of chlorine-resistant finishes, and their use for this purpose has been patented [168]. The methyl, ethyl, and hydroxyethyl triazones (based respectively on methylamine, ethylamine, and ethanolamine) have received most attention and it has been suggested that the buffering powers of the tertiary amine groups should be sufficient to neutralise any hydro-chloric acid liberated on ironing, if any chlorine has been retained by the treated fabric. There are disadvantages in commercial products, however, as the reaction producing the triazones is reversible and consequently they may contain free amine, free formaldehyde, or methylolureas. Careful aftertreatment is therefore essential to prevent development of fishy odours and to enable the tertiary amine group to protect the fabric fully from chlorine retained by the urea–formaldehyde component. Triazones are very weak bases and the mechanism whereby they are said to protect the fabric from acid damage therefore appears to be somewhat unsatisfactory. The efficiency of these products as a basis for chlorine-resistant finishes has been discussed by several authors [169–172], and their use in conjunction with urea–formaldehyde has been described [173], while a series of triazones prepared from long-chain amines has recently been examined [174].

There is a structural relationship between the triazones and melamine which was recently discussed by Marsh [175], and it is not surprising that other compounds related to melamine have also received attention. A paper by Vail et al.[176] describes work carried out with acetoguanamine (XIII) and a number of N-substituted melamines, of which NN'N"-trimethylmelamine (XXII) is of particular interest.
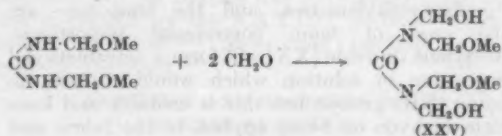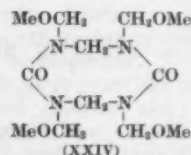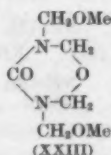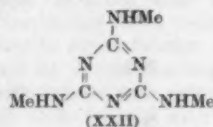
The Urons

The reaction producing dimethyloluron (VIII), already mentioned, is the basis of a number of patents [177], but there is still some doubt about the existence of this compound which, in any event, is stable only in solution and readily loses form-aldehyde. A derivative, dimethoxymethyluron (XXIII), was described by Kadowaki [178], and may

form the basis of new chlorine-resistant finishes recently described [179]. The nature of commercial materials is uncertain, however, and they may well contain less formaldehyde and include compounds such as tetramethoxymethyldimethylenediureide (XXIV), which was also prepared by Kadowaki, and should be equally resistant to chlorine.

A recent patent [180] has proposed the use of dimethoxymethyldimethylolurea (XXV) prepared in a similar way to the urons, but the existence of this compound would again appear doubtful.

NON-NITROGENOUS CROSS-LINKING AGENTS FOR CELLULOSE

It can be appreciated that much research has been carried out in an effort to find a crease-proofing agent completely unaffected by chlorine, and an obvious solution to the problem is to employ a nitrogen-free compound as a cellulose cross-linking agent. Epoxy compounds and glycol acetals have been extensively investigated, but their creaseproofing efficiency is inferior to that of the amino resins. Nevertheless, the chemistry of these products is of considerable interest.

Epoxy Resins

The use of epoxy resins to creaseproof cellulosic fabrics was first suggested some years ago [181] and has been discussed in a number of recent American publications. Commercial epoxy resins are usually of the diglycidyl ether type (XXVI) and are only partially soluble in water, so that an emulsion system is required for their application to textiles. Diepoxy compounds which are soluble in water have now been developed, and one of the more promising of these is vinyl-cyclohexene dioxide (XXVII).

$$CH_2-CH-CH_2-O-R-O-CH_2-CH-CH_2$$
$$\underset{O}{\diagdown}\diagup \qquad\qquad \underset{O}{\diagdown}\diagup$$
(XXVI)
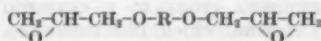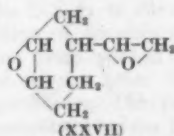
$$\begin{array}{c} CH_2 \\ CH \;\; CH-CH-CH_2 \\ O \;\;|\;\;\;\; | \;\;\;\; O \\ CH \;\; CH_2 \\ CH_2 \end{array}$$
(XXVII)

The use of such compounds in textile finishing has been discussed by Schroeder and Condo [182], who employed a commercial resin (Eponite 100) in emulsified form and zinc fluoroborate catalyst. Work on a variety of epoxy derivatives has recently been described by Steele [183]. The relatively high price of the epoxides and the toxicity of the catalyst have tended to discourage extensive commercial development, and later work [159, 184, 185] has been concerned with the use of combinations of epoxy resins with the cyclic urea types of cross-linking agents. Progress in this field has recently been discussed [186].

Glycol Acetals

Another type of non-nitrogenous cross-linking agent for cellulose [187] is based on the reaction of formaldehyde with a compound containing two or more hydroxyl groups, the product from ethylene glycol and formaldehyde, e.g., having the structure—
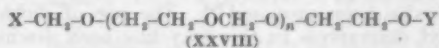
$$X-CH_2-O-(CH_2-CH_2-OCH_2-O)_n-CH_2-CH_2-O-Y$$
(XXVIII)

Application of products of this type is very effective in the dimensional stabilisation of cellulosic fabrics [188], but the crease recovery of the treated fabric is relatively poor. Better results are obtainable with a product based on the reaction of formaldehyde with pentaerythritol (XXIX).
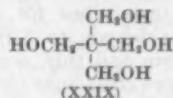
$$\begin{array}{c} CH_2OH \\ | \\ HOCH_2-C-CH_2OH \\ | \\ CH_2OH \end{array}$$
(XXIX)

Subsequent work in this field has been described [189, 190], and a summary of the present position has been published [191]. It would again appear that the creaseproofing efficiency of this type of product leaves something to be desired, particularly when smooth-drying properties are required, so that the use of polyacetals in conjunction with cyclic urea derivatives has been examined [156].

#### Dialdehydes and Bischloromethyl Ethers

The use of glyoxal as a creaseproofing agent was examined some considerable time ago, and the possibility of using dialdehydes of various chain lengths to obtain cellulose cross-links of different flexibilities is of considerable theoretical interest. Products of this type have been suggested in the patents literature [192] and the effects obtained have been described [193]. Unfortunately, the possibility of acetal formation by reaction of one aldehyde group with two molecules of cellulose cannot be excluded, so that the cross-links formed may be either of type A or of type B.

$$\begin{array}{cc} Cell-O\cdot CH_2-R-CH_2O-Cell & Cell-O-CH-O-Cell \\ & | \\ & R \\ & | \\ & CHO \\ \qquad A & \qquad B \end{array}$$
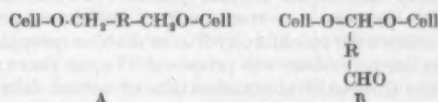
The possibility of cross-linking cellulose by means of the bischloromethyl ether of ethylene glycol (XXX) has also been suggested [194]. The use of compounds of this type in the form of their pyridinium derivatives, e.g. XXXI, has recently been investigated [195].

$$Cl\cdot CH_2\cdot O\cdot CH_2\cdot CH_2\cdot O\cdot CH_2\cdot Cl$$
(XXX)
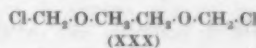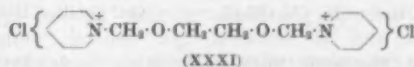
$$Cl\left\{ \langle \rangle \overset{+}{N}\cdot CH_2\cdot O\cdot CH_2\cdot CH_2\cdot O\cdot CH_2\cdot \overset{+}{N} \langle \rangle \right\} Cl$$
(XXXI)

The chloromethyl ether is prepared by reaction of the glycol, formaldehyde, and hydrochloric acid at low temperature, and the pyridinium derivative is then obtained by reaction of the product with pyridine. It is clear that a range of materials of this type can be prepared by varying the structure of the glycol employed; Tesoro [155] examined several compounds. Reaction of bischloromethyl ethers with cellulose produces polyformal cross-links similar to those obtained from the glycol acetals

mentioned above. Combination of the process with the application of synthetic rubber latices has also been proposed [196]. The use of dichloro-alkyl derivatives in this way has been discussed by Marsh [197].

Formaldehyde (Dry or Wet Cross-linking)

Treatment of cellulose with formaldehyde has been practised for many years and formed the basis of the "sthenosage" process for dimensional stabilisation of viscose rayon fabrics. Unfortunately, the conditions for successful cross-linking by formaldehyde to form a methylene-cellulose are severe and this, combined with the rigidity of the short cross-links obtained, results in a severe reduction in fabric strength. The reaction has been intensively studied in recent years, several papers [198-201] being of particular interest. The subject has been reviewed by Roff [202], who has also carried out some experimental work [203] in this field; a comprehensive paper has been published by Marsh [204]. Information on the rate of reaction of formaldehyde with cellulose at various temperatures has been obtained by Datye and Nabar [205].

Recent work in the U.S.A. has been directed towards the treatment of cellulose in a partially swollen condition with formaldehyde. Guthrie [206] has shown that cotton fabrics having excellent wet crease recovery can be obtained in this way, while Reeves et al.[207] have suggested a commercial method of applying the process and have compared treatments involving fully or partly swollen cellulose [208].

Other Wet Cross-linking Processes

It is possible for cellulose to react in the swollen state with cross-linking agents other than form-aldehyde; one of these forms the basis of the recently developed Belfast process [209]. The basis of this process is a treatment of cellulose in the wet state with epichlorohydrin or dichloropropanol. A similar procedure was proposed [210] some years ago for the dimensional stabilisation of cotton fabrics, and this was referred to by Bouvier [6] in 1955 as a creaseproofing process. It does, in fact, effect a remarkable improvement in wet crease recovery, but the dry crease recovery is, if anything, diminished by the process, which also reduces considerably the tear and tensile strengths of the fabric. The reaction proceeds as follows—

$$CH_2Cl \cdot CHCl \cdot CH_2OH + NaOH \longrightarrow CH_2Cl \cdot CH\underset{O}{-}CH_2 + NaCl + H_2O$$

$$Cell \cdot OH + CH_2\underset{O}{-}CH \cdot CH_2Cl \longrightarrow Cell \cdot O \cdot CH_2 \cdot CHOH \cdot CH_2Cl$$

$$Cell \cdot O \cdot CH_2 \cdot CHOH \cdot CH_2Cl + NaOH \longrightarrow Cell \cdot O \cdot CH_2 \cdot CH\underset{O}{-}CH_2 + NaCl + H_2O$$

$$Cell \cdot O \cdot CH_2 \cdot CH\underset{O}{-}CH_2 + HO \cdot Cell \longrightarrow Cell \cdot O \cdot CH_2 \cdot CHOH \cdot CH_2 \cdot O \cdot Cell$$

Modern developments of the process include a dry treatment with a cyclic urea type of cross-linking agent before or after the treatment with dichloropropanol to improve both dry and wet crease recovery.

A further wet cross-linking process which has recently aroused considerable interest is based on the treatment of a cellulosic fabric with divinyl sulphone (XXXII) and alkali. The Remazol type of reactive dyes discussed below form vinyl sulphones which react with cellulose under alkaline conditions in a similar way.

$$\begin{array}{c} CH_2:CH \\ | \\ SO_2 \\ | \\ CH_2:CH \\ (XXXII) \end{array}$$

$$-SO_2 \cdot CH:CH_2 + HO-Cell \rightarrow -SO_2 \cdot CH_2 \cdot CH_2 \cdot OCell$$
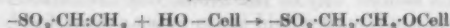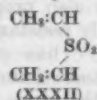
Divinyl sulphone was first proposed by Welch and Guthrie [211] as a means of linking a dye to cellulose, while its use for cross-linking two cellulose molecules was patented some time ago [212]. Its use for this purpose has recently been investigated by Tesoro et al.[213], following an earlier suggestion by Reeves et al.[214]. The toxicity of divinyl sulphone, which is related to mustard gas (dichloro-diethylsulphide) in structure, was a major disadvantage, and less toxic derivatives based on earlier work by Stahmann [215] were employed by Tesoro et al.

Miscellaneous Topics

RELATION BETWEEN DYES AND RESINS

The improvement in washing fastness of dyeings obtained by the application of amino resins to cellulosic fabrics was appreciated in the early days of the process, and numerous relevant patents appeared. The introduction of the concept of cellulose cross-linking in recent years suggests that a direct chemical combination of suitable dyes with cellulose should be possible; developments have now taken place in this field, resulting in the appearance of the reactive dyes. The Procion and Cibacron reactive dyes are obtained by attaching the chromophoric group to a cyanuric chloride (XXXIII) residue, which, like melamine, contains the triazine ring structure.

$$\begin{array}{c} Cl \\ | \\ C \\ N \diagdown \quad \diagup N \\ || \qquad | \\ Cl \diagup C \diagdown_{N} \diagup C \diagdown Cl \\ (XXXIII) \end{array}$$
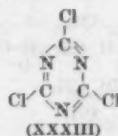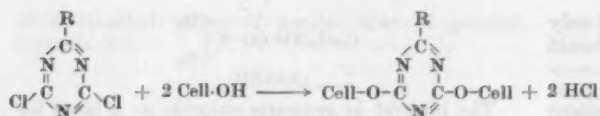
One or two of the chlorine atoms in this compound are replaced by suitable chromophoric groups, and the resultant dyes can be caused to react with the hydroxyl groups of cellulose under alkaline conditions to give a dyeing of exceptional washing fastness. The Remazol dyes contain a group such as $-SO_2 \cdot CH_2 \cdot CH_2 \cdot Cl$ or $-SO_2 \cdot CH_2 \cdot CH_2 \cdot SO_3H$ attached to the dye residue and, under alkaline conditions, a vinyl sulphone ($-SO_2 \cdot CH:CH_2$) is formed which is capable of reacting with cellulose as shown above. Although chemical combination with cellulose is possible in all these cases, cross-links between cellulose molecules are only possible with dyes containing the dichlorotriazine residue.

$$\underset{Cl}{\overset{R}{\underset{N}{\overset{C}{\underset{N}{\overset{\|}{C}}}}}} \quad + \ 2\ Cell\text{-}OH \longrightarrow Cell\text{-}O\text{-}C \quad C\text{-}O\text{-}Cell + 2\ HCl$$

The reactions involved in the application of Procion or Cibacron dyes [216, 217] and of Remazol dyes [218, 219] have been described. In addition to the above reaction with cellulose, it is possible for reactive dyes to combine with wool or nylon and with amino resins. A process based on the simultaneous application of Procion dyes and amino resins to cellulosic fabrics has recently been developed [220], using a special catalyst to facilitate reaction. A process using alkaline catalysts has also been investigated [221].

It has long been known that some dyes are affected by formaldehyde; direct dyes in particular should be chosen with care if the fabric is to be resin-finished. A further problem which has been extensively investigated is the effect of resin-finishing processes on the light fastness of dyeings. This can be improved in some cases, but there are many examples of dyeings which are reduced in light fastness by resin treatments. In recent years the effect of resins on the light fading of direct [222] and vat [223] dyes has been examined. The general problem has recently been investigated [224] and the behaviour of the reactive dyes has been discussed [225-227]. It appears to be generally agreed that finishes based on DMEU have a particularly adverse effect on the fastness of reactive dyes, while the smallest effect is observed in the case of dimethylol-dihydroxyethyleneurea, although this marked difference in behaviour has not been satisfactorily explained.

PERMANENT CREASING AND THE CREASEPROOFING OF GARMENTS

There has been some interest in recent years, particularly in the U.S.A., in developing processes for the introduction of durable creases into garments made from resin-treated fabrics and the creaseproofing of fabrics after these have been made up into garments. The process suggested for permanent creasing involves hydrolysis of the resin finish followed by re-curing by hot pressing, and finishes based on DMEU are recommended for this purpose. Investigations in this field have been reported by several groups of workers [228-231].

The creaseproofing of garments which have not previously been resin-treated has also been examined [232, 233], and here again finishes based on DMEU have been preferred. Processes of this type present obvious difficulties, and the possibility of fishy odours developing during the treatment or on subsequent storage of the garments cannot be excluded, since curing conditions are likely to be inadequate and an after-wash is impracticable.

RESIN APPLICATIONS OTHER THAN CREASEPROOFING

The various uses of amino resins not directly connected with the production of smooth-drying and chlorine-resistant finishes have received relatively little attention in recent literature. The use of melamine resins in the form of acid colloids for stiffening cellulosic fabrics, and urea or thiourea products in nylon stiffening, has already been mentioned. The dimensional stabilisation of fabrics composed of various fibres has been fully discussed [234]. The use of chemical finishing agents in the stabilisation of knitted cotton fabrics has been reviewed [235]; the particular characteristics of knitted fabrics render special processing techniques necessary, if satisfactory results are to be obtained on resin finishing. Numerous patents have appeared relating to the development of new durable mechanical finishes, but these refer principally to new techniques for the application of conventional amino resins and the production of new effects by modification of the mechanical finishing stage of the process. Specialised products of the amino-resin type are also concerned in the considerable advances which have been made in the field of pigment printing. Solvent-soluble resins in emulsion form are employed for this purpose; recent developments have been described [236, 237]. Improvements in pigment fixation have been concerned mainly with the quality of the pigments employed and the evolution of binder formulations having the minimum adverse effect on the handle of the fabric.

DEVELOPMENT OF FISHY ODOURS

One of the problems associated with resin finishing has been the development of fishy odours in the treated fabric on storage, particularly under humid conditions. This has always been associated with the presence in the fabric of catalyst residues and by-products of the condensation process; washing-off after the baking operation has usually been advocated to remove them. Rising production costs have rendered the washing-off process uneconomic and it is frequently omitted, particularly when metal-salt catalysts are employed. The problems associated with the development of fishy odours have been discussed [238] and suggestions have recently been made [239] concerning the substances in the resin bath which are responsible for them. It has been shown in particular that formate must be present, and the use of resins from which formate has been removed by ion exchange appears to prevent the development in the treated fabric of methylamines, which are considered to be responsible for the fishy odours. Since formate is always present in commercial resins, owing to the presence of formic acid in technical formalin, it follows that this factor is always present and the avoidance of other contributory factors, e.g. presence of free formaldehyde and ammonium salts, assumes much greater importance. These factors are reduced to a minimum in methylated urea–formaldehyde precondensates, which are probably more widely used than the ammonia-stabilised type at present. The replacement of ammonium salt catalysts by magnesium chloride or zinc nitrate is also of considerable value in this respect. It should be emphasised that hot, humid conditions are particularly conducive to odour development and fabrics which are to be Hofmann pressed or exported to
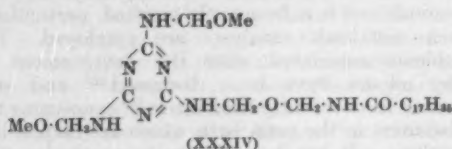
tropical countries should be resin-treated only under the best possible conditions and should preferably be washed-off.

WATER-REPELLENT AND OIL-REPELLENT FINISHES
Long-chain Paraffin Derivatives

The development of a water-repellent finish durable to washing and dry-cleaning has aroused particular interest in recent years. This period has coincided with the commercial development of the silicones and most of the published work has been concerned with these products. The other main basis for durable water-repellency has been the fatty acid amides, and stearamide derivatives of various kinds have been frequently mentioned in the patent literature. The whole field has recently been reviewed [240].

With the development of resin finishes, an interest arose in the possible adaptation of the process to give durable water-repellency. One suggestion which has persisted even in comparatively recent patents has been to apply urea-formaldehyde [241] or melamine–formaldehyde [242] pre-condensates in conjunction with a paraffin wax emulsion. There would not appear to be adequate justification, however, for assuming that the durability of a wax emulsion finish could be improved in this way. The incorporation of a fatty chain in the resin molecule would appear more promising, and finishes based on etherification of melamine resins with fatty alcohols or esterification with fatty acids have been proposed [243]. The modification of urea or melamine resins by reaction with methylolstearamide was also suggested at a relatively early date, and compounds such as XXXIV are thought to be involved, while a similar product based on DMEU and methylol stearamide has recently been patented [244].

$$NH \cdot CH_2OMe$$
$$C$$
$$N \quad N$$
$$C \qquad C \quad NH \cdot CH_2 \cdot O \cdot CH_2 \cdot NH \cdot CO \cdot C_{17}H_{35}$$
$$MeO \cdot CH_2NH \qquad N$$
(XXXIV)

It is not essential that a resin component should be present if water repellency alone is desired, and compounds such as methylolstearamide (XXXV) and its pyridinium derivative (XXXVI), which are capable of reacting with cellulose, were suggested more than twenty years ago.

$$C_{17}H_{35}CO \cdot NH \cdot CH_2OH \qquad C_{17}H_{35}CO \cdot NH \cdot CH_2 \cdot \overset{+}{N} \bigcirc \Big\} Cl$$
(XXXV)   (XXXVI)

Recent developments in this field include the improvement of pyridinium derivatives of methylolstearamide [245] and the production of more stable dispersions of methylolstearamide itself by using stearamide containing a reduced proportion of free fatty acid [246]. Other patents refer to methylolamides [247], the use of amino resins in conjunction with stearamidomethoxyacetic acid [248] or stearatochromic chloride [249], the stearylamine salt of stearylcarbamic acid [250], and the octadecyl derivative of asymmetrical ethyleneurea [251] (XXXVII).

$$C_{18}H_{37}NH \cdot CO-N \overset{\displaystyle CH_2}{\underset{\displaystyle CH_2}{|}}$$
(XXXVII)
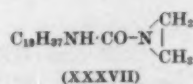
The interest in cyanuric chloride as a basis for dyes capable of reacting with cellulose has a parallel in the field of water-repellent agents. It has been suggested, e.g., that a water-repellent finish can be obtained by joint application of a fatty amine and cyanuric chloride to a cellulosic fabric [252], while application of 2-stearylamino-4,6-dichloro-1,3,5-triazine (XXXVIII) has also been proposed [253].

$$NH \cdot C_{17}H_{35}$$
$$C$$
$$N \quad N$$
$$Cl \quad C \qquad C \quad Cl$$
$$N$$
(XXXVIII)
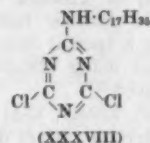
Silicones

Considerable progress has been made with the introduction of silicone-based water-repellent agents [254], which can be applied in conjunction with amino-resin finishes [255]. Fully methylated silicones or dimethylpolysiloxanes require very severe curing conditions, while methyl hydrogen siloxanes, which are more reactive, tend to affect the handle of the fabric adversely. A satisfactory practical compromise can be reached by a combination of both types.

Patent developments in this field have been numerous, but are related principally to minor modifications or the development of suitable catalysts and will not be discussed here. The literature contains many references to silicone water-repellent agents and the subject had been discussed extensively both in this country and in the U.S.A.[256-264]; in one paper [257], silicones are compared with stearamide-based water-repellent agents for the treatment of cellulose acetate. A recent paper by Conner et al.[265] is of interest, since they suggest a new type of silicone formulation. This consists of a blend of tetravinyl silane (XXXIX) polymer with methyl hydrogen siloxane polymer, which they describe as a "silicone alloy" and for which they claim finishes of improved durability.

$$CH:CH_2$$
$$CH_2:CH-Si-CH:CH_2$$
$$CH:CH_2$$
(XXXIX)
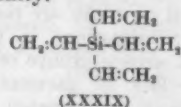
Fluorochemicals

As water-repellent agents, neither the silicones nor the stearamide-based products can be regarded as the final answer to the problem of durability and for this we must look to future developments. These may well be concerned with the fluorochemical finishes which have recently come into prominence and for which both durable water-repellency and oil-repellency are claimed [266]. Work has recently been reported on various types of product in this field, and chromium complexes of perfluoromonocarboxylic acids have been examined [267, 268]. Segal et al.[269] employed polymers

of fluoroalkyl esters of acrylic acid of general formula XL—

$$\left[\begin{array}{c} CH_2-CH \\ | \\ COOCH_2R_f \end{array} \right]_n$$

(XL)
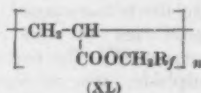
where R_f is a perfluorinated alkyl group.

The durability of finishes based on commercial fluorochemicals is not fully satisfactory, and recent American work has suggested that improved results are obtained with combinations of this type of product with the older materials based on stearamide [270]. Further work by Benerito *et al.*[271] is of some technical interest, although the process they have employed does not appear to be readily adaptable to commercial development. By reaction of perfluorobutyryl chloride or perfluorooctanoyl chloride in dimethylformamide solution with small samples of cotton fabric, they have prepared the corresponding perfluoro-esters of cellulose and have found that the treated fabrics show water-repellency and oil-repellency of excellent durability. A recent paper [272] summarises previous work in this field and presents the results of a comprehensive examination of formulations based on fluorochemicals in conjunction with amino resins, stearamide-based water-repellent agents and silicones. The advantages of using fluorochemicals in combination with stearamide-based water-repellent agents have recently been emphasised [273].

The use of water-repellent agents as additives to amino-resin finishes to improve the tear strength, sewability, and abrasion resistance of the treated fabric has been mentioned earlier in this paper. Both stearamide-based products and silicones have been examined for this purpose; the stearamide-modified amino resins, in particular, contribute towards the crease recovery of the treated fabric and enable the quantity of creaseproofing agent to be reduced, while the silicones appear to assist the smooth-drying properties of the material. The quantities of these additives employed for softening purposes are less than those required for the production of water-repellency, and the silicones can be used without the addition of a special silicone catalyst.

FLAMEPROOFING OF CELLULOSIC FABRICS

Earlier processes for flameproofing cellulosic fabrics were based on water-soluble materials, e.g. borax and ammonium phosphate, and were completely non-durable. The production of flameproof finishes of limited durability followed and it is only recently that an approach to a fully durable and adequately flameproof fabric could be achieved. Public interest in this problem has been increasing in recent years and has led to questions in Parliament and to the issue of a British Standard for flameproof fabrics (*BS* 3121). Unfortunately, a partial solution to the problem of durability is of little value, since a flameproof garment which loses its non-inflammable properties after a few washes will then become a serious potential danger to the purchaser.

A review of this subject must necessarily cover many processes which have been proposed, but which have never been commercially successful for a variety of reasons. They are of some interest, nevertheless, as they show the wide variety of chemicals which have been investigated in this connection. The majority of the compounds proposed are phosphorus derivatives of one type or another, while halogen and antimony compounds have also been suggested. A number of reviews of the flameproofing field have appeared in recent years. Several authors [274-277] have discussed the general theory of flameproofing of cellulose, while the behaviour of the cellulose–phosphate–urea complex, which forms the basis of some of the earlier processes, has been considered [278]. Richards [279] has recently published a general review of modern flameproofing processes, while Frieser has reviewed the flameproofing of both cellulosic [280] and synthetic-polymer [281] textiles, and applications to military fabrics have been discussed by McQuade [282]. Wraight and Thomas [283] have recently described experiments in which various methods of testing the inflammability of fabrics were compared.

Earlier processes based on amino resins and phosphoric acid [284] probably involved the phosphorylation of cellulose under controlled conditions. The treated fabric contained phosphoric acid groups neutralised with ammonia and, on washing, these ammonium groups were replaced by sodium and the fabric lost its flameproof properties. Other patented processes involve the application of condensation products of ammonia and phosphoryl chloride [285], halogenated phosphoric esters [286], derivatives of ammonia and phosphonitrilic chloride [287], phosphonitrilic chloride polymers [288], phosphonitrilic esters [289], esters of phosphorodiamidic acid [290], and melamine–formaldehyde resins in conjunction with phosphoramidates [291]. It is probable that none of these processes has been developed commercially.

One process which has led to significant commercial development is that based on the application to cellulosic fabrics of tetrakishydroxymethylphosphonium chloride or THPC (XLI) in conjunction with a melamine resin [292].

$$\left. \begin{array}{c} CH_2OH \\ | \\ HO \cdot CH_2-P^+-CH_2OH \\ | \\ CH_2OH \end{array} \right\} Cl^-$$

(XLI)
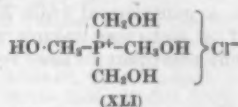
After application to the fabric, the finish is dried and heat cured in the usual manner and, since hydrochloric acid is liberated during this process, THPC formulations contain urea as a buffering agent as well as the resin precondensate. A number of patents relating to this process have now appeared [293] and its application has been discussed in some detail [294-296]. The distribution of phosphorus in fabrics treated with THPC has also been investigated [297].

The possibility of using various derivatives of THPC, including a product of its reaction with ethyleneimine, has also been suggested in the patent literature [298], but it is unlikely that such processes are employed commercially. The disadvantages of the original THPC process, which
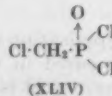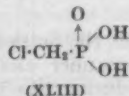
involves a high-temperature baking operation and can affect fabric strength adversely in the way that other resin-based finishes do, have stimulated research on alternative application procedures. A recent announcement [299] suggests that improved results may be obtained by a new THPC process which does not include a high-temperature baking operation, but details of the method have not yet been published.

The combination of phosphorus and bromine in flameproof finishes has also been extensively investigated, and the patent literature contains references to brominated triallyl phosphate polymers [300] and bromine-containing nitrilophosphorus compounds [301]. The use of formulations based on a reaction product of bromoform and triallyl phosphate has been discussed by Frick et al.[302], while Reid et al.[303] have examined its use in conjunction with THPC. The application of brominated phosphonitrilates alone [304] or as additives to THPC [305] has also been reported; replacement of bromine by other halogens has been discussed [306].

Several other methods of introducing phosphorus into cellulosic fabrics have also been examined. Work on phosphorylamide, described in patents [307], has been reported by Nielsen [308]. The structure of this compound, which is produced by the reaction of ammonia with phosphoryl chloride, is unknown, but it is a highly water-soluble polymer containing the group XLII, and is applied by the normal impregnation and curing technique. It forms the basis of the commercial Flame Retardant PA (Monsanto), but the durability of this type of product has proved disappointing.

$$\begin{array}{c} O \\ \uparrow \\ -P-NH- \\ | \\ NH_2 \end{array}$$
(XLII)
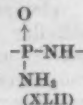
Another process of considerable technical interest is the phosphonomethylation of cotton [309]. The reaction is carried out by treating the fabric with chloromethylphosphonic acid (XLIII) in presence of sodium hydroxide, or with chloromethylphosphoryl chloride (XLIV). The application of the process on a commercial scale has been discussed [310], and a wet-cross-linking pretreatment based on dichloropropanol has recently been proposed [311].

$$\begin{array}{cc} O & O \\ \uparrow \diagup OH & \uparrow \diagup Cl \\ Cl\cdot CH_2\cdot P & Cl\cdot CH_2\cdot P \\ \diagdown OH & \diagdown Cl \\ (XLIII) & (XLIV) \end{array}$$

Another phosphorus compound which has been extensively examined is trisaziridinylphosphine oxide or APO (XLV). This material is prepared by reaction of ethyleneimine with phosphorus oxychloride, and it has been found capable of producing on cotton fabrics a creaseproof finish resistant to chlorine.

$$\begin{array}{cc} \begin{array}{c} H_2C\!-\!CH_2 \\ \diagdown N \diagup \\ H_2C \diagdown \quad | \quad \diagup CH_2 \\ \quad |\ N\!-\!P\!-\!N\ | \\ H_2C \diagup \quad \downarrow \quad \diagdown CH_2 \\ \quad O \end{array} & \begin{array}{c} CH_2OH \\ | \\ HO\cdot CH_2\!-\!P\!\rightarrow\!O \\ | \\ CH_2OH \end{array} \\ (XLV) & (XLVI) \end{array}$$
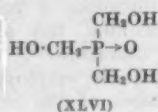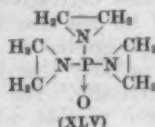
Reeves et al.[312] found that it reacted with THPC; they applied the reaction product to cotton fabrics and obtained excellent flameproof finishes. The commercial possibilities of this process have recently been discussed [313]. The use of APO and the corresponding sulphide, APS, in conjunction with THPC or trishydroxymethylphosphine oxide (XLVI) has recently been patented [314]. The use of APO in creaseproofing cotton fabrics has been described by Drake and Guthrie [315], while Miles et al.[316] have examined the infrared spectra of treated cellulose and have shown that APO can be used successfully for flameproofing without addition of THPC, if diammonium phosphate is used as catalyst.

Methods of imparting flameproof properties to cotton by direct chemical modification have been discussed by Pacsu and Schwenker [317]. They investigated the mesylation and tosylation of cellulose by treatment with methanesulphonyl chloride or toluene sulphonyl chloride, respectively, using the method proposed by Roberts [318]. This procedure did not produce adequately flameproof material, but substitution of methane sulphonyl groups by bromine or iodine by treatment with the corresponding sodium halide gave promising results. Further improvement was achieved by subsequent phosphorylation of the cellulose, using a reaction analogous to mesylation based on treatment with diethyl chlorophosphonate (cf. ref. 286). It is probable that means such as this produce the most durable flameproof finishes, but, since the reaction is carried out in pyridine solution and is relatively slow, commercial application of the process in its present form is unlikely.

An entirely different approach to the flameproofing problem depends on the application of antimony compounds to the fabric. The basis of certain processes of this type is the use of antimony oxide, Sb_2O_3, in conjunction with a binding agent. Patented processes suggest the use of chlorinated polyvinyl chloride [319], a combination of an amino resin and polyvinyl chloride [320], or the latter in admixture with a chlorine-containing plasticiser [321]. Commercial processes of this type have been discussed by Read and Heighway-Bury [322].

Commercial flameproof finishes are at present expensive owing to the very high quantities of flameproofing agent which are required and the high cost of such materials. The handle of the fabric is also affected adversely and the problem of providing a reasonably priced and fully durable finish remains to be solved.

ROTPROOFING OF CELLULOSE AND PRODUCTION OF BACTERICIDAL FINISHES

Attack of cellulosic fabrics by moulds and bacteria, particularly under hot and humid conditions has always been a problem, and rotproofing processes of a non-durable type have long been established. The evolution of equally effective treatments of a durable nature has been extensively investigated in recent years and several interesting developments have taken place. It was appreciated at an early stage that the crease-resist process imparted a degree of rot-resistance to cellulosic fabrics, particularly when phenolic resins were employed. Phenol–formaldehyde precondensates

are as effective as urea–formaldehyde in crease-proofing, but the discoloration of the fabric following their use was an obstacle to commercial development.   For certain applications a rot-proofing agent which discolours the fabric is still acceptable, and some recent work has described the use of halogenated phenolic resins in the production of rot-resistant fabrics.   Bromo-phenol–formaldehyde [325] and chlorophenol–form-aldehyde [324] precondensates have been examined.

The use of melamine–formaldehyde resins in rot-proofing has been reviewed by Cooke [325], who employed conventional methylated methylol-melamines.   Although the resin alone showed appreciable rotproofing properties in soil-burial tests, the best results were obtained in conjunction with a recognised rotproofing agent, 8-hydroxy-quinoline (XLVII), the mixture showing advantages over either component used alone.

(XLVII)
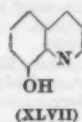
A process based on an acid colloid solution pre-pared from melamine resin and excess of formic acid has recently been described [326] and its possible commercial application discussed [327].   This proce-dure uses considerably greater quantities of formic acid than are required for the preparation of the conventional type of melamine acid colloid used in shrinkproofing wool, and the stability of the resin bath is relatively poor.   The process has been compared [328] with the Arigal process of Ciba [329], which consists in the condensation of melamine and formaldehyde in the wet fabric by storage under acid conditions.

A further development in recent years is the production of bactericidal finishes for wearing apparel; this subject has been discussed [330, 331]. It was hoped, in the first instance, that a bactericide could be rendered durable by application to the fabric in conjunction with an amino resin.   Un-fortunately, chemical combination with the resin renders the bactericide ineffective, and if combina-tion does not take place the resultant finish is not durably bactericidal.   The same difficulty is encountered if attempts are made to produce a durable insecticidal fabric by treatment with an amino resin in conjunction with, e.g., DDT or dieldrin, although insecticidal coatings on imper-vious surfaces can be produced in this way by using a lacquer or paint based on a solvent-soluble amino resin containing the insecticide in solution [332]. Crystallisation of the insecticide on the coated surface occurs in this case and satisfactory results are obtained.   The most recent developments in the field of bactericides have involved the treatment of fabrics with neomycin, which is substantive to the fibres and is applied by a process analogous to dyeing [333].   Results obtained in extensive trials of this process have been described by Salsbury [334].

It is hoped that this survey will suffice to show the considerable advances in resin-finishing tech-niques in recent years and the wide variety of new products which have been investigated.   As can be seen, old processes have been adapted to the production of much improved finishes and a wide variety of chemicals has achieved a new promi-nence.   The future holds many possibilities and it can be expected that even greater technical advances in this field will be achieved in the next few years.

*   *   *

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References

[1] Smith, A. R., J.S.D.C., **70**, 381 (1954).
[2] Borghetty, *Text. World*, **108**, No. 4, 89 (1958).
  Fortess, *Amer. Dyestuff Rep.*, **47**, 513 (1958).
  Gutmann, J.S.D.C., **75**, 81 (1959).
  Pieper, *Rayonne fibres synth.*, **14**, 785 (1958).
  Sookne, *Amer. Dyestuff Rep.*, **49**, 890 (1960).
[3] Marsh, *J. Textile Inst.*, **48**, P370 (1957).
[4] Best-Gordon, J.S.D.C., **69**, 41 (1953).
[5] Best-Gordon, Pieper, and Tattersfield, *Rayonne fibres synth.*, **11**, 493 (1955).
[6] Bouvier, *Industr. text.*, 765 (Oct 1955).
[7] Borghetty and Fornelli, *Text. World*, **105** (No. 12), 102 (1955).
[8] Foster, *Text. Research J.*, **27**, 129 (1957).
[9] Smith, A. R., *ibid.*, **26**, 836 (1956).
[10] Tovey, *ibid.*, **31**, 185 (1961).
[11] Borghetty, *Amer. Dyestuff Rep.*, **47**, 509 (1958).
[12] Marsh, *Text. Manuf.*, **84**, 577 (1958); **86**, 202 (1960); J.S.D.C., **76**, 23 (1960).
[13] Nuessle, *Text. Industr.*, 116 (Oct 1959).
[14] Enders, *Melliand Textilber.*, **40**, 1315, 1454 (1960).
[15] Cronin, *Amer. Dyestuff Rep.*, **49**, 389 (1960).
[16] British Industrial Plastics (BIP), *BP* 767,789; *USP* 2,820,715.
[17] BIP, *BP* 787,674.
[18a] De Jong, *Rec. Trav. chim.*, **69**, 1566 (1950).
  [18b] De Jong and de Jonge, *ibid.*, **71**, 643, 661, 890 (1952).
  [18c] De Jong, de Jonge, and Eden, *ibid.*, **72**, 88 (1953).
  [18d] De Jong and de Jonge, *ibid.*, **72**, 139, 202, 207, 213, 653, 1027 (1953).
[19] Landqvist, *Acta Chem. Scand.*, **9**, 1459 (1955).
[20] Dunn, *J.C.S.*, 1446, 4687 (1957).
[21] Glauert, *Ind. Chemist*, **33**, 392 (1957).
[22] Steele and Giddings, *Text. Research J.*, **26**, 116 (1956); *Ind. Eng. Chem.*, **48**, 110 (1956).
[23] Steele and Schiwall, *Text. Research J.*, **29**, 228 (1959).
[24] Mehta and Mehta, *ibid.*, **30**, 524 (1960).
[25] Steele, *ibid.*, **31**, 489 (1961).
[26] Eapen, Modi, and Mehta, *ibid.*, **31**, 181 (1961).
[27] Douglas, J.S.D.C., **73**, 258 (1957).
[28] American Cyanamid Co. (ACY), *BP* 804,291.
  Fishman, *Amer. Dyestuff Rep.*, **44**, 403 (1955).
[29] ACY, *USP* 2,881,154.
[30] ACY, *USP* 2,881,153.
[31] ACY, *BP* 856,466.
  Ciba, *BP* 822,929.
[32] ACY, *USP* 2,881,152.
[33] Nickerson, *Text. Research J.*, **27**, 54 (1957).
[34] ACY, *BP* 679,386.
  Monsanto, *USP* 2,661,313.
[35] ACY, *BP* 680,623; *USP* 2,539,366.
[36] BIP, *BP* 727,660; 730,872.
[37] Monsanto, *BP* 711,477.
  Bradford Dyers' Assocn., *BP* 734,587.
[38] Beer, *BP* 687,856; 689,102.
[39] ICI, *BP* 790,796.
  Van Bochove and Schuringa, *Teintex*, **19**, 505 (1954).
[40] ACY, *BP* 727,425.

[42] Ciba, *BP* 730,018.
[43] Perkins and Reeves, *Text. Research J.*, **30**, 305 (1960).
[44] Chance, Perkins, and Reeves, *ibid.*, **30**, 918 (1960).
[45] Cooke, Dusenbury, Kienle, and Lineken, *ibid.*, **24**, 1015 (1954).
[46] Nuessle, Fineman, and Heiges, *ibid.*, **25**, 24 (1955).
[47] Lineken, Davis, and Jorgensen, *ibid.*, **26**, 940 (1956).
[48] Landells and Whewell, J.S.D.C., **72**, 137 (1956).
Landells, Leathley, and Whewell, *ibid.*, **73**, 496 (1957).
[49] Gardon and Steele, *Text. Research J.*, **31**, 160 (1961).
[50] Steiger, Wang, and Hurwitz, *ibid.*, **31**, 327 (1961).
[51] Mehta and Mehta, *ibid.*, **31**, 182 (1961).
[52] Gagliardi and Shippee, *ibid.*, **31**, 316 (1961).
[53] Marsh, *Text. Manuf.*, **85**, 294, 373, 557 (1959).
[54] O'Brien and van Loo, *Text. Research J.*, **31**, 340 (1961).
[55] Perkerson, Reeves, and Tripp, *ibid.*, **30**, 944 (1960).
[56] Reeves, Perkins, and Chance, *ibid.*, **30**, 179 (1960).
[57] Frick, Andrews, and Reid, *ibid.*, **30**, 495 (1960).
[58] Tripp, Moore, and Rollins, *ibid.*, **31**, 295 (1961).
[59] Williams, *Amer. Dyestuff Rep.*, **48** (No. 13), 27 (1959).
[60] Morton and Beaumont, J.S.D.C., **76**, 578 (1960).
[61] Steele, *Text. Research J.*, **30**, 37 (1960).
[62] Rath, Herbolsheimer, and Stapf, *ibid.*, **30**, 201 (1960).
[63] Marsh, *Text. Manuf.*, **85**, 35 (1959).
[64] White, J.S.D.C., **70**, 284 (1954).
[65] Enders, *Melliand Textilber.*, **38**, 547 (1957).
Enders and Zorkendorfer, *ibid.*, **40**, 1076 (1959); **41**, 462 (1960).
[66] Park, J.S.D.C., **76**, 624 (1960).
[67] Tripp, Moore, de Gruy, and Rollins, *Text. Research J.*, **30**, 140 (1960).
[68] Aenishaenslin, *S.V.F. Fachorgan.*, **12**, 602 (1957).
[69] Marsh, *Text. Rec.*, **78**, 72 (May 1960); 58 (June 1960); 63 (July 1960).
[70] Orr, Burgis, de Luca, and Grant, *Text. Research J.*, **31**, 302 (1961).
Orr, Burgis, and Grant, *ibid.*, **31**, 550 (1961).
[71] Rebenfeld, *ibid.*, **31**, 311 (1961).
[72] Lippert, *ibid.*, **26**, 128 (1956).
[73] Steele, *ibid.*, **26**, 739 (1956).
[74] Sookne, Krasny, Hart, and Scott, *Amer. Dyestuff Rep.*, **49**, 457 (1960).
[75] AATCC, Rhode Island Section, *ibid.*, **44**, 779 (1955).
[76] Wannow, *Melliand Textilber.*, **38**, 1276 (1957).
[77] Tootal Broadhurst Lee Co. (TBL), *BP* 727,881–2; 727,888–90.
Courtaulds, *BP* 818,919; 825,608.
[78] Mazzeno *et al.*, *Amer. Dyestuff Rep.*, **46**, 719 (1957).
[79] Reinhardt, Kullmann, Moore, and Reid, *ibid.*, **47**, 758 (1958).
[80] TBL, *BP* 486,926; 527,761; 557,067.
Perlmuteer, *BP* 490,135.
[81] ICI, *BP* 518,916.
[82] TBL, *BP* 712,098.
[83] TBL, *BP* 695,703.
[84] Joseph Bancroft and Sons Co., *BP* 742,885.
[85] Linen Industry Research Assocn., *BP* 766,265.
[86] Abrams and Sherwood, *Amer. Dyestuff Rep.*, **42**, 777 (1953).
[87] AATCC, South-Eastern Section, *ibid.*, **43**, 780 (1954).
[88] Sherwood, *ibid.*, **44**, 262 (1955).
[89] Steele and Browne, *ibid.*, **45**, 525 (1956).
[90] Rosenbaum, *ibid.*, **48** (No. 10), 46 (1959).
[91] Mazzeno, Markezich, and Reid, *ibid.*, **47**, 299 (1958).
[92] AATCC, Piedmont Section, *ibid.*, **43**, 41 (1954); **44**, 141 (1955).
[93] Simpson, *ibid.*, **46**, 991 (1957); *Text. Research J.*, **28**, 170 (1958).
[94] Marsh, *Text. Manuf.*, **86**, 449 (1960).
[95] Quehl, *BP* 703,432.
[96] Monsanto, *BP* 660,366; 705,608.
[97] Kingsley and Pianka, *BP* 709,078.
[98] Ciba, *BP* 716,952.
[99] Joseph Bancroft and Sons Co., *BP* 720,016.
[100] Boehme Fettchemie, *BP* 767,249.
[101] ICI, *BP* 841,640.
[102] TBL, *BP* 827,645–6.
[103] Bleachers' Assocn., *BP* 846,287.
[104] Irvine, Simons, and Davids, *Amer. Dyestuff Rep.*, **48** (No. 12), 37 (1959).
[105] Gagliardi, *ibid.*, **45**, 99 (1956).
[106] Marsh, *Text. Manuf.*, **86**, 70, 113 (1960).

[107] Cooke, *Amer. Dyestuff Rep.*, **43**, 891 (1954).
Cooke *et al.*, *ibid.*, **46**, 585 (1957); *Text. Research J.*, **27**, 150 (1957).
[108] Steele, *J. Applied Polymer Sci.*, **4**, 45 (July–Aug 1960).
[109] Ziifle, Berni, and Benerito, *Text. Research J.*, **31**, 349 (1961).
[110] TBL, *BP* 704,143; 713,001–2; 728,440.
Linen Industry Research Assocn., *BP* 750,002–3; 758,391.
[111] Alvsaker and Turner, J.S.D.C., **71**, 797 (1955).
[112] Foster, *Text. Research J.*, **26**, 149 (1956).
[113] Kaswell, *Amer. Dyestuff Rep.*, **48** (No. 7), 56 (1959).
[114] Hunter and Lofland, *ibid.*, **48** (No. 8), 54 (1959).
[115] Cluett and Peabody Inc., *Text. Merc.*, **143**, 559 (1960).
[116] Sloan *et al.*, *Amer. Dyestuff Rep.*, **49**, 302 (1960).
[117] Ernst and Braschler, *Textil-Rund.*, **14**, 512 (1959).
[118] TBL, *BP* 846,875.
[119] Williams, *Amer. Dyestuff Rep.*, **45**, 472 (1956).
[120] Bercaw, *ibid.*, **49**, 111 (1960).
[121] Williams, *ibid.*, **48** (No. 4), 37 (1959).
[122] Sudnik, *J. Textile Inst.*, **51**, T227 (1960).
[123] Sudnik, *Text. Research J.*, **31**, 78 (1961).
[124] AATCC, Washington Section, *Amer. Dyestuff Rep.*, **48** (No. 21), 57 (1959).
[125] Linen Industry Research Assocn., *BP* 743,300.
[126] Stiegler *et al.*, *Amer. Dyestuff Rep.*, **45**, 685 (1956).
[127] Cooke, *ibid.*, **47**, 679 (1958).
[128] Briar, *ibid.*, **49**, 559 (1960).
[129] Breens and Morton, J.S.D.C., **71**, 513 (1955).
[130] Best-Gordon and Morton, *ibid.*, **71**, 874 (1955).
[131] Bacon, Parker, and Horn, *Amer. Dyestuff Rep.*, **46**, 933 (1957).
[132] Reid, Arceneaux, Reinhardt, and Harris, *ibid.*, **49**, 490 (1960).
[133] Reid, Reinhardt, Kullman, and Arceneaux, *ibid.*, **49**, 527 (1960).
[134] Arceneaux, Gautreaux, Reinhardt, and Reid, *ibid.*, **50**, 163 (1961).
[135] Wachter, Davison, and Knight, *ibid.*, **42**, 261 (1953).
[136] Wham and Mack, *ibid.*, **42**, 285, 328, 360, 389, 455 (1953).
Wham and Lynn, *ibid.*, **42**, 421 (1953).
[137] AATCC Tentative Test Method 69–52, *AATCC Year Book*, p. 96 (1953).
[138] AATCC, *Amer. Dyestuff Rep.*, **48** (No. 1), 5; (No. 16), 51 (1959).
[139] Schefer, *Textil-Rund.*, **12**, 512 (1957); **14**, 17, 587 (1959).
[140] Walker, *Formaldehyde* (New York: Reinhold Publishing Corpn.), 2nd Edn., p. 301 (1953).
[141] Howard, *Text. Research J.*, **26**, 124 (1956).
[142] Rümens, *S.V.F. Fachorgan.*, **13**, 10 (1958).
[143] Smith, J. E., Bacon, and Hughes, *Amer. Dyestuff Rep.*, **47**, 259 (1958).
[144] Nuessle, *ibid.*, **47**, 465 (1958).
[145] Reinhardt, Mazzeno, Fenner, and Reid, *ibid.*, **47**, 505 (1958).
[146] Mazzeno, Kullman, Reinhardt, and Reid, *ibid.*, **47**, 609 (1958).
[147] Mosher, *Text. Research J.*, **29**, 88 (1959).
[148] Smith, A. R., *Amer. Dyestuff Rep.*, **48** (No. 8) 49 (1959).
[149] Frick, Kottes, and Reid, *Text. Research J.*, **29**, 314 (1959).
[150] Mehta and Mody, *ibid.*, **30**, 532 (1960).
[151] AATCC, Piedmont Section, *Amer. Dyestuff Rep.*, **49**, 843 (1960).
[152] Schweitzer, *J. Org. Chem.*, **15**, 471 (1950).
[153] ICI, *BP* 575,260; 577,735.
Joseph Bancroft and Sons Co., *BP* 690,237.
[154] Stam, Poon, Spangler, and Underwood, *Text. Research J.*, **26**, 960 (1956).
[155] Walter, Buxbaum, and Green, *ibid.*, **27**, 146 (1957).
[156] Marsh, *Text. Manuf.*, **84**, 527 (1958); **87**, 153 (1961).
[157] Dan River Mills Inc., *USP* 2,690,404.
Jennings, *Text. Research J.*, **25**, 20 (1955).
[158] Frick *et al.*, *Amer. Dyestuff Rep.*, **48** (No. 18), 37 (1959).
[159] Gagliardi and Shippee, *Text. Research J.*, **29**, 54 (1959).
[160] Enders and Pusch, *Melliand Textilber.*, **49**, 314, 429 (1959); *Amer. Dyestuff Rep.*, **49**, 1 (1960).
[161] Cooke and Roth, *Text. Research J.*, **29**, 180 (1959).
[162] Frick, Kottes, and Reid, *Amer. Dyestuff Rep.*, **48** (No. 13), 23 (1959).
[163] Marsh, *Text. Rec.*, **77**, 63 (Feb 1960).
[164] TBL, *BP* 831,297.

165 BASF, *BP* 717,287; 720,386–7; *USP* 2,731,364.
Phrix-werke, *BP* 771,328.
166 Rümens, Götz, and Zeidler, *Melliand Textilber.*, **41**, 330 (1960); *Amer. Dyestuff Rep.*, **49**, 752 (1960).
167 Paquin, *Angewandte Chem. A.*, **60**, 267 (1948).
168 Sun Chemical Corpn., *BP* 845,468.
169 Wayland, *Text. Research J.*, **29**, 170 (1959).
170 Marsh, *Text. Manuf.*, **85**, 187 (1959).
171 Reid, Frick, Reinhardt, and Arceneaux, *Amer. Dyestuff Rep.*, **48** (No. 3), 81 (1959).
172 Goldstein, *ibid.*, **48** (No. 5), 44 (1959).
173 Frick, Arceneaux, Reinhardt, and Reid, *ibid.*, **48** (No. 15), 29 (1959).
174 Vail, Frick, Murphy, and Reid, *ibid.*, **50**, 200 (1961).
175 Marsh, *Text. Manuf.*, **87**, 25 (1961).
176 Vail, Frick, and Reid, *Amer. Dyestuff Rep.*, **50**, 356 (1961).
177 DuP, *USP* 2,373,135; 2,485,203; 2,452,200.
178 Kadowaki, *Bull. Chem. Soc. Japan*, **11**, 248 (1936).
179 Dexter Chemical Co., *Textile World*, **110**, 70 (Aug 1960).
180 DuP, *USP* 2,825,710.
181 N.V. de Bataafsche Petroleum Maatschappij, *BP* 732,573.
Shell Chemical Corpn., *USP* 2,752,269.
182 Schroeder and Condo, *Text. Research J.*, **27**, 135 (1957).
183 Steele, *ibid.*, **31**, 257 (1961).
184 McKelvey, Webre, and Benerito, *Amer. Dyestuff Rep.*, **49**, 804 (1960).
185 Galligan *et al.*, *Text. Research J.*, **30**, 208 (1960).
186 Marsh, *Text. Rec.*, **77**, 69 (Nov 1959).
187 Quaker Chemical Products Corpn., *BP* 776,478; 780,043; *USP* 2,785,947–9; *USP* 2,785,996–7; 2,786,081.
188 Irvine and Kress, *Text. Research J.*, **28**, 148 (1958).
189 Irvine, Cronin, and Davids, *ibid.*, **29**, 74 (1959).
190 Kress, *Amer. Dyestuff Rep.*, **48** (No. 4), 33 (1959).
191 Marsh, *Text. Rec.*, **77**, 136 (Aug–Sept 1959).
192 Phrix-werke, *BP* 710,441; 727,919.
Algemene Kunstzijde Unie, *BP* 721,380.
193 Hurwitz and Conlon, *Text. Research J.*, **28**, 257 (1958).
194 Etab. Schaeffer et Cie., *BP* 783,382.
195 Tesoro, *Text. Research J.*, **30**, 192 (1960).
196 Traitements Chimiques des Textiles, *BP* 837,508.
197 Marsh, *Text. Rec.*, **78**, 52 (Dec 1960).
198 Steele, *Text. Research J.*, **25**, 545 (1955).
199 Kärrholm, *ibid.*, **25**, 756 (1955).
200 Woo, Dusenbury, and Dillon, *ibid.*, **26**, 745, 761 (1956).
201 O'Brien and van Loo, *ibid.*, **31**, 276 (1961).
202 Roff, *J. Textile Inst.*, **49**, T646 (1958).
203 Roff, *ibid.*, **50**, T353 (1959).
204 Marsh, J.S.D.C., **75**, 244 (1959).
205 Datye and Nabar, *Text. Research J.*, **30**, 72 (1960).
206 Guthrie, *ibid.*, **29**, 834 (1959).
207 Reeves, Perkins, and Chance, *Amer. Dyestuff Rep.*, **49**, 639 (1960).
Chance, Perkins, and Reeves, *Text. Research J.*, **31**, 71 (1961).
208 Chance, Perkins, and Reeves, *ibid.*, **31**, 366 (1961).
209 Deering Milliken Research Corpn., *BP* 855,547.
210 Fothergill and Harvey (Research and Development), *BP* 696,282; 724,096; 767,991.
cf. Marsh, *Text. Rec.*, **78**, 52 (Dec 1960).
211 Welch and Guthrie, *Text. Research J.*, **31**, 84 (1961).
212 United States Rubber Co., *USP* 2,524,399.
213 Tesoro, Linden, and Sello, *Text. Research J.*, **31**, 283 (1961).
214 Reeves, Drake, McMillan, and Guthrie, *ibid.*, **25**, 41 (1955).
215 Stahmann *et al.*, *J. Org. Chem.*, **11**, 719 (1946).
216 Vickerstaff, J.S.D.C., **73**, 237 (1957).
217 Dawson, Fern, and Preston, *ibid.*, **76**, 210 (1960).
218 Bohnert, *ibid.*, **75**, 581 (1959).
219 Luttringhaus, *Amer. Dyestuff Rep.*, **50**, 248 (1961).
220 *Text. Weekly*, **60** (1671), 729 (1960).
ICI, *Tech. Inf. Dyehouse No. 546, No. 580*; *BP* 846,505.
Rattee and Bugmann, *S.V.F. Fachorgan Textilver.*, **16**, 162 (1961).
221 AATCC, Delaware Valley Section, *Amer. Dyestuff Rep.*, **49**, 810 (1960).
222 AATCC, Rhode Island Section, *ibid.*, **43**, 6 (1954).
223 AATCC, Piedmont Section, *ibid.*, **45**, 821 (1956); **47**, 39 (1958).
224 Goldstein and Koenig, *Text. Research J.*, **29**, 66 (1959).

225 Glarum, *Amer. Dyestuff Rep.*, **48** (No. 5), 41 (1959).
226 Marsh, *Text. Manuf.*, **85**, 465 (1959).
227 ICI, *Tech. Inf. Dyehouse No. 510*.
Ciba Rev., No. 135, 41 (1959).
228 Reid, Mazzeno, Reinhardt, and Markezich, *Text. Research J.*, **27**, 252 (1957).
229 Reid, Reinhardt, and Kullman, *ibid.*, **28**, 242 (1958).
230 Graham, Loibl, and Wiebush, *ibid.*, **28**, 252 (1958).
231 Reeves and Mack, *Amer. Dyestuff Rep.*, **48** (No. 21), 43 (1959).
232 Hurwitz, *Text. Research J.*, **27**, 267 (1957).
233 Graham, *ibid.*, **27**, 270 (1957).
234 Matlin and Nuessle, *Ind. Eng. Chem.*, **47**, 1729 (1955).
235 Irvine, *Text. Research J.*, **30**, 222 (1960).
236 Nelson, *Amer. Dyestuff Rep.*, **48** (No. 1), 1 (1959).
237 Cassel, *ibid.*, **49**, 184 (1960).
238 Marsh, *Text. Rec.*, **76**, 85 (Dec 1958).
239 Nuessle, Heiges, and Olney, *Text. Research J.*, **29**, 41 (1959).
240 Krause, *Z. ges. Textilind.*, **62**, 882, 990 (1960).
241 Nico ter Kuile & Zonen, *BP* 692,900.
242 Ciba, *BP* 727,015.
243 Ciba, *BP* 709,333; 709,386.
244 BIP, *BP* 865,319.
245 ICI, *BP* 713,756.
246 ICI, *BP* 835,596.
247 General Aniline, *BP* 679,811; *USP* 2,692,837; 2,693,430.
248 ICI, *BP* 748,182.
249 ICI, *BP* 767,834.
250 FBy, *BP* 767,945.
251 FH, *BP* 799,045.
252 Calico Printers' Assocn. (CPA), *BP* 818,778.
253 CPA, *BP* 818,942.
254 Dow Corning, *BP* 686,212.
Midland Silicones, *BP* 753,730.
255 ICI, *BP* 763,464.
256 Payne and Smith, E., *Brit. Rayon & Silk J.*, **30**, 91 (Oct 1953).
257 Mann, J.S.D.C., **71**, 579 (1955).
258 Watt, *J. Textile Inst.*, **48**, T175 (1957); **51**, T1 (1960).
259 Madaras, J.S.D.C., **74**, 835 (1958).
260 Gibson and Dennett, *Amer. Dyestuff Rep.*, **42**, 275 (1953).
261 Fortess, *Ind. Eng. Chem.*, **46**, 2325 (1954).
262 Cook and Shane, *Text. Research J.*, **25**, 105 (1955).
263 Ford, *Amer. Dyestuff Rep.*, **45**, 377 (1956).
264 Cook, *ibid.*, **45**, 771 (1956).
265 Conner, Reeves, and Chance, *Text. Research J.*, **30**, 171 (1960).
266 Minnesota Mining and Mfg. Co., *BP* 823,974; *USP* 2,622,835; 2,642,416; *Text. Bull.*, **82** (No. 10), 115 (1956).
Grajeck and Petersen, *Amer. Dyestuff Rep.*, **48** (No. 13), 37 (1959).
Enders and Wiest, *Textil-Praxis*, **15**, 1293 (1960).
Bradford Dyers' Assocn., *BP* 858,022.
267 Philips, Segal, and Loeb, *Text. Research J.*, **27**, 369 (1957).
268 Benerito, Berni, and Fagley, *ibid.*, **30**, 393 (1960).
269 Segal, Philips, Loeb, and Clayton, *ibid.*, **28**, 233 (1958).
270 De Marco *et al.*, *Mod. Text.*, **41**, 50 (1960).
271 Benerito, Berni, and Fagley, *Text. Research J.*, **30**, 576 (1960).
272 AATCC, Rhode Island Section, *Amer. Dyestuff Rep.*, **49**, 818 (1960).
273 Goldstein, *Text. Research J.*, **31**, 377 (1961).
274 Schuyten, Weaver, and Reid, *Ind. Eng. Chem.*, **47**, 1433 (1955).
275 Ward, J.S.D.C., **71**, 569 (1955).
276 Gottlieb, *Text. Research J.*, **26**, 156 (1956).
277 Sandholzer, *Amer. Dyestuff Rep.*, **48** (No. 2), 37 (1959).
278 Nuessle, Ford, Hall, and Lippert, *Text. Research J.*, **26**, 32 (1956).
279 Richards, *Canadian Textile J.*, **77** (No. 26), 68 (1960).
280 Frieser, *Melliand Textilber.*, **39**, 795, 1034 (1958).
281 Frieser, *ibid.*, **40**, 436 (1959).
282 McQuade, *Amer. Dyestuff Rep.*, **44**, 749 (1955).
283 Wraight and Thomas, *J. Textile Inst.*, **51**, T203 (1960).
284 ACY, *BP* 690,291; *USP* 2,582,961.
Ciba, *BP* 747,014; 772,367.
Gy, *BP* 790,663.
285 Monsanto, *BP* 683,572; 700,822.

[286] Glenn L. Martin Co., *BP* 688,372.
Celanese Corpn. of America, *BP* 694,217.
[287] Francolor, *BP* 774,694.
[288] Albright & Wilson, *BP* 788,785.
[289] U.S. Secretary of Agriculture, *USP* 2,681,295.
[290] Bleachers' Assocn., *BP* 799,606.
[291] Courtaulds, *BP* 835,581.
[292] Reeves and Guthrie, *Text. Merc.*, 130, 474 (1954);
Dyer, 111, 567 (1954).
[293] Albright & Wilson, *BP* 740,269; 790,641.
Bradford Dyers' Assocn. and Albright & Wilson,
BP 761,985.
[294] Guthrie, Drake, and Reeves, *Amer. Dyestuff Rep.*, 44,
328 (1955).
[295] Reeves and Guthrie, *Ind. Eng. Chem.*, 48, 64 (1956).
[296] Perfect, J.S.D.C., 74, 829 (1958).
[297] Stansbury and Hoffpauir, *Amer. Dyestuff Rep.*, 44,
645 (1955).
[298] Albright & Wilson, *BP* 764,313–4; 821,504.
[299] Proban Ltd., *Text. Merc.*, 142, 653 (1960).
[300] Glenn L. Martin Co., *BP* 700,292; 700,455; 700,604.
[301] Albright & Wilson, *BP* 807,236.
[302] Frick, Weaver, and Reid, *Text. Research J.*, 25, 100
(1955).
[303] Reid, Frick, and Arceneaux, *ibid.*, 26, 137 (1956).
[304] Hamalainen and Guthrie, *ibid.*, 26, 141 (1956).
[305] Hamalainen, Reeves, and Guthrie, *ibid.*, 26, 145 (1956).
[306] Frick, Reid, and Moore, *ibid.*, 26, 525 (1956).
[307] Monsanto, *USP* 2,661,264; 2,661,311; 2,661,342.
[308] Nielsen, *Text. Research J.*, 27, 603 (1957).
[309] Drake, Reeves, and Guthrie, *ibid.*, 29, 270 (1959).
[310] Hobart, Drake, and Guthrie, *ibid.*, 29, 884 (1959).
[311] *Idem, Amer. Dyestuff Rep.*, 50, 92 (1961).
[312] Reeves *et al.*, *Text. Research J.*, 27, 260 (1957).
[313] Drake, Beninate, and Guthrie, *Amer. Dyestuff Rep.*,
50, 129 (1961).
[314] Albright & Wilson, *BP* 837,709–10.
[315] Drake and Guthrie, *Text. Research J.*, 29, 155 (1959).
[316] Miles, Hofmann, and Merola, *Amer. Dyestuff Rep.*, 49,
596 (1960).
[317] Pacsu and Schwenker, *Text. Research J.*, 27, 173 (1957);
Schwenker and Pacsu, *Ind. Eng. Chem.*, 50, 91 (1958).
[318] Roberts, *J. Amer. Chem. Soc.*, 79, 1175 (1957).
[319] Quehl, *BP* 747,569.
[320] Courtaulds, *BP* 800,157.
[321] Associated Lead Manufacturers, *BP* 791,802–3.
[322] Read and Heighway-Bury, J.S.D.C., 74, 823 (1958);
Texture, 5, 85 (June 1958).
[323] Bell, Ramsey, and Whewell, J.S.D.C., 71, 660 (1955).
[324] Chance, Perkerson, and McMillan, *Text. Research J.*,
29, 558 (1959).
[325] Cooke, *ibid.*, 24, 197 (1954).
[326] Berard, Gautreaux, and Reeves, *ibid.*, 29, 126 (1959).
[327] McMillan *et al.*, *Amer. Dyestuff Rep.*, 49, 180 (1960).
[328] Ruperti, *Text. Research J.*, 30, 68 (1960).
[329] Ciba, *BP* 745,638; 820,946; *USP* 2,763,574; *Ciba
Rund.*, No. 108, 3985 (1953).
[330] Sherrill, *Text. Research J.*, 26, 342 (1956).
[331] Yeager, *Canadian Textile J.*, 76 (No. 26), 42 (1959).
[332] National Research Development Corpn., *BP* 709,721;
Dyte, *Pest Technology*, 98 (Feb. 1960).
[333] ACY, *BP* 788,968; 842,217.
[334] Salsbury, *Mod. Text. Mag.*, 38 (No. 4), 82 (1957).

Discussion

Mr. S. M. JAECKEL: With regard to chlorine-retention, if the concentration of DMEU applied were raised, could this lead to greater loss of tensile strength on chlorination owing to a greater amount of self-condensation as well as cellulose cross-linkage?

Mr. SMITH: Laboratory experiments suggest that the opposite is the case and that chlorine-retention is more likely to occur when low concentrations of DMEU are applied. This effect is counteracted to some extent by increasing the concentration of catalyst but cannot be fully overcome and is one of the reasons for the gradual replacement of DMEU by more satisfactory types of chlorine-resistant finish.

Mr. JAECKEL: Although amino-resin finishes were developed for creaseproofing, they can actually lead to the formation of creases on prolonged storage of the fabric if the cure was originally incomplete. Are any resins better or worse in this respect?

Mr. SMITH: This effect undoubtedly occurs, particularly with spun rayons, and it is not possible to recommend any particular resin as better in this respect. Adequate curing is essential, but even if attainable, a fabric completely resistant to pressing would not be satisfactory for garment production.

Mr. J. WARDLE: Can the speaker give any comments on reasons for "yellowing"?

Mr. SMITH: "Yellowing" or discoloration of resin-treated fabrics can be produced by heat alone in some cases and has been observed after curing the fabric when formulations incorporating certain thermoplastic latices, e.g. butadiene–acrylonitrile, have been employed. Finishes based on "triazones" can discolour in the same way. Yellowing after treatment with hypochlorite solutions is characteristic of melamine resins and is dependent on the number of free –NH– groups left after curing. It is said to be reduced by use of magnesium chloride as catalyst[107] and should be eliminated when derivatives of hexamethylol-melamine are employed[109].

Mr. McDONALD: Reference has been made to the possibility of cellulose cross-links being formed from more than one molecule of DMEU and it has been suggested that intermediate ether linkages are susceptible to hydrolysis and give rise to chlorine retention on storage of the treated fabric under humid conditions. Is it not true that methylene links rather than $-CH_2-O-CH_2-$ links are formed between DMEU molecules, which would not break down as suggested?

Mr. SMITH: It is true that methylene links are formed under strongly acid conditions, e.g. at pH 2–3, but under neutral conditions ether linkages are produced. Under normal curing conditions, say at pH 5, it is probable that both forms of linkage exist and sufficient ether linkages will certainly be present to account for the practical effects observed.

Mr. JAECKEL: There are numerous analytical schemes for resin identification, ranging from dyeing and colour reactions to chromatography. Which does the speaker prefer?

Mr. SMITH: Experience has shown that none of the proposed analytical schemes are fully satisfactory, particularly when mixtures of resins of different types have been employed. It is essential to compare colour reactions with those of fabric samples treated in a known manner; even then, intermediate and ambiguous results are frequently obtained. The same considerations also apply to chromatography and I can only recommend the use of all test methods available together with a considerable amount of experience in analysis of resin finishes. The source of supply of the test fabric and the type of finish involved will often provide valuable clues as to the type of resin likely to be present.

Colour-matching Lamps— An Enquiry into Members' Wishes

K. McLaren

A committee of the British Standards Institution is at present engaged in revising the obsolete specification for daylight-matching lamps, *BS* 950:1941, and the Society of Dyers and Colourists is represented on this committee by the author of this report. A detailed study of all the relevant factors has revealed many convincing reasons for basing such lamps on the spectral distribution of total daylight, i.e. sunlight plus skylight, rather than on north-sky daylight, which has traditionally been used by professional colour matchers. It has also shown that there is no valid reason for basing colour-matching lamps on Illuminant C, the principal source in colorimetry, which formed the basis of *BS* 950:1941. A critical review of all the factors relevant to the subject of illumination in colour matching will be published in the *Journal* in the near future.

This subject is of such great importance to members of the Society that Council thought that they should be given the opportunity of expressing their views. Therefore in March 1961 the following questionnaire was sent to all members and subscribers to the *Journal* and to other interested organisations, e.g. the Textile Institute and the Society of Leather Trades' Chemists.

Please indicate your preference with an X

1.   Colour-matching lamps should imitate—

    North-sky daylight

    Total daylight (i.e. sunlight + skylight)

2.   If north-sky daylight is preferred, which curve should be chosen?

    Illuminant C

    Abbot–Gibson 7400°K

Any observations on any aspects of colour-matching lamps will be welcome.

This questionnaire was accompanied by an explanatory note which is reproduced as an Appendix.

By the end of June, 171 replies had been received. These have been analysed, with the following result—

Question 1

    71   selected north-sky daylight
    98   selected total daylight
    2   made no choice but made comments
    ───
    171

Question 2

Of the individuals who selected north-sky daylight in Question 1

    15   chose Illuminant C
    54   chose the Abbot–Gibson 7400°K curve
    2   made no choice
    ──
    71

Although the questionnaire and the accompanying notes stated quite clearly that a choice between Illuminant C and the Abbot–Gibson curve could only logically be made by those who preferred north-sky daylight, 12 individuals who chose total daylight also answered Question 2. In three cases a logical reason for so doing was given: two individuals said that, although they preferred total daylight, should the consensus of opinion favour north-sky daylight, then they felt that the Abbot–Gibson curve would be a more suitable choice than Illuminant C; the third said that lamps imitating total daylight and conforming to the Abbot–Gibson curve were both necessary "because of the quantity of near-whites which must be matched by bleachers and makers-up"; the other 9 individuals (8 of whom chose the Abbot–Gibson curve and 1 Illuminant C) gave no reasons for the illogical voting and one is left with the suspicion that they had not fully understood the basic issues involved.

These results can be summarised as follows—

A majority, 58%, of the members who replied preferred total daylight to north-sky daylight as a basis for colour-matching lamps (55·6% if the votes of those who voted illogically are discounted); only 9% of voting members were in favour of perpetuating Illuminant C for this purpose.

56 Individuals accepted the opportunity of making observations on aspects of colour-matching lamps. Many merely explained the reasons for their voting choice or stressed the need for standardisation in this field, but the following other aspects were frequently mentioned, these being arranged in order of popularity, the most popular being mentioned first—

(1)   Provision should be made for inspection under tungsten light as well as standard daylight to detect metameric matches.

(2)   Some means of detecting when the quality of the light has changed significantly (with age, by soiling, or deterioration of any reflectors in the lamp) is necessary.

(3)   The light must be of adequate intensity and be emitted from a large area.

(4)   It would be preferable if retail stores would also use the colour-matchers' standard light for display purposes.

(5)   The xenon arc (colour temperature 6000–6500°K) should be established as the standard.

The replies to the questionnaire have been of considerable help to the author of this report in formulating the Society's considered views on daylight-matching lamps and Council expresses its thanks to all members who co-operated.

Appendix
EXPLANATORY NOTE

The colour-rendering properties of any light source are defined precisely by its spectral-energy distribution, that is, by the proportion of the total energy emitted by the lamp at each wavelength in the visible spectrum. The energy distribution in the ultraviolet region is also important where fluorescent colours or fluorescent brightening agents (optical bleaches) are present; the lack of sufficient ultraviolet radiation is a common failing among some present daylight lamps and should be rectified in a new specification.

There is no such thing as an "ideal" spectral energy distribution for detecting small colour differences, except possibly an equal-energy light source, which would radiate equal amounts of energy at all wavelengths between specified limits. However, such a light source is of academic interest only, since it does not correspond to any natural light.

A *standard* light source which could be universally accepted would, however, be of great value. Many of the problems of colour matching arise from the existence of metameric pairs, that is, pairs of patterns which appear to have the same colour under one light but no longer match under another type of illumination. The existence of a standard reference lamp would minimise disputes as to whether two patterns are an acceptable match, although such disputes would not be eliminated, since differences in the colour vision of individual observers can be as important as differences in illumination.

The purpose of an "artificial daylight lamp" is to imitate some aspect of daylight, and the main difficulty arises in defining the kind of daylight which is to be imitated. It is obvious that the colour of the light coming from a brilliant blue sky is different from that from a grey, overcast sky, while the light in late evening is different from that at mid-day. Discussion of the different phases of daylight is simplified by the use of "colour temperatures". When any substance, such as a piece of iron, is heated in the dark, it first becomes visible as a dull red colour, which then changes to orange and finally to white, or bluish white, as the temperature is raised. The colour of the light can therefore be defined in terms of the temperature of the source— the bluer the light, the higher the temperature.

The natural light normally used by professional colourists is that coming from a north-facing window or skylight, but even here the colour temperature varies widely, from about 5000°K on a very dull day with an overcast sky to over 15,000°K on a clear day with a blue sky. North-sky daylight is chosen by colourists not so much for its colour, but because it is fairly constant in intensity, direct sunlight being intermittent and glaring.

On the other hand, in everyday use, coloured materials are viewed under illuminations which differ even more widely in colour temperature, from ordinary tungsten light at about 2600°K to north-sky daylight at over 15,000°K. In addition, modern developments in fluorescent and discharge lighting introduce further complications, as the spectral-energy distributions of these lights are different from any phase of daylight or tungsten light. Naturally, the special conditions of viewing under artificial light do not directly affect the specification of an "artificial *daylight* lamp". It is probably true to say that the most common conditions under which the majority of coloured materials are viewed is "total daylight", which is a combination of light coming directly from the sun together with light coming from the rest of the sky.

Surprisingly enough, the colour quality of total daylight is fairly constant with a colour temperature of about 6000°K. Such light is just as good as any phase of north-sky daylight for detecting small colour differences, and its more constant colour makes it much superior to the north-sky light used by colour matchers. If a metameric match cannot be avoided, then, if the patterns are arranged to match under a lamp conforming to the spectral-energy distribution of total daylight, they will still match under most daylight conditions. Furthermore, total daylight has a colour temperature which lies in the middle of the wide range of colour temperatures encountered in daily life.

The first question to be answered, therefore, is whether an artificial daylight lamp should be designed to imitate either (a) total daylight or (b) north-sky light. If a decision is made to imitate total daylight, the specification should be relatively simple. Total daylight has the same spectral distribution as sunlight outside the atmosphere, and there is a proposed standard curve of such radiation (Moon, *J. Franklin Inst.*, 230, 583 (1940)); this curve extends into the ultraviolet, and lamps conforming to this distribution would be suitable for the visual examination of fluorescent material. The colour temperature of this light is 6200°K.

On the other hand, if north-sky daylight is preferred, then there are two spectral-energy distribution curves which might be imitated by an artificial daylight lamp. These are, first, the standard (Illuminant C) established by the International Commission on Illumination (C.I.E.) in 1931, which has a colour temperature of 6740°K, and, second, a distribution computed by Abbot and Gibson as representing a moderately overcast north-sky daylight with a colour temperature of 7400°K. The chief merits and demerits of these two possibilities can be briefly summarised as follows—

Illuminant C
Advantages
(1) It is a widely used standard source for colorimetry, and is likely to remain so for many years.

Defects
(1) It is seriously deficient in ultraviolet radiation as compared to any phase of daylight,
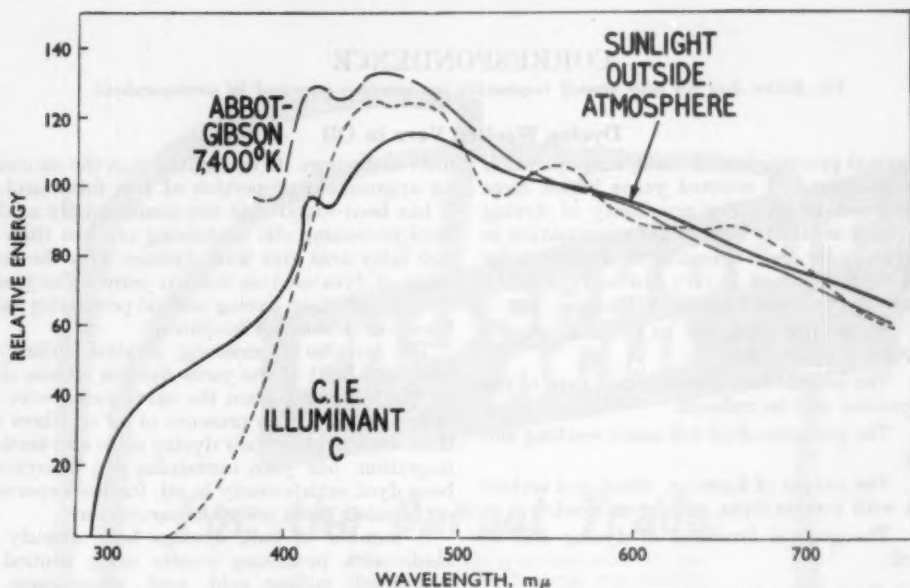
and cannot be used for the inspection of fluorescent materials.

(2) The spectral-energy distribution of Illuminant C is determined by a set of liquid filters used in conjunction with a tungsten filament lamp. Although this illuminant was intended to represent north-sky light, the actual energy distribution is determined partly by the liquid filters employed, and there seems to be no cogent reason why a daylight lamp, which almost certainly will not employ liquid filters, should be required to duplicate this particular curve.

Abbot–Gibson 7400° K

Advantages

(1) The energy distribution defined by this curve has been found to approximate more closely than Illuminant C to a moderately overcast north-sky daylight in the U.S.A., and this is probably true also in the U.K. and in other countries.

(2) The curve is computed from measurements of solar radiation and not of a particular light source; although at present it extends down to only 380 millimicrons (mμ), it could probably be extended to 300 millimicrons, which would define the ultraviolet characteristics for the examination of fluorescent specimens.

(3) The curve has been adopted as a standard for colour-matching lamps in the U.S.A.

Defects

(1) No convenient tables are yet available whereby spectrophotometric curves could be converted into colorimetric data representing the appearance of samples under this illuminant. It is doubtful, however, whether differences between the calculated values obtained from the Abbot–Gibson curve and those obtained from Illuminant C would be of practical significance.

The three spectral-energy distribution curves discussed are illustrated in the diagram.

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Dyeing Woollen Yarn in Oil

It is normal practice before the dyeing operation to scour woollen and worsted yarns which have been processed in oil. The possibility of dyeing woollen yarns in oil has been under examination in this laboratory for some time and the results so far obtained are promising. If yarn can be dyed in the oiled state, certain advantages will accrue, e.g.—

(1) Processing costs will be reduced, since a yarn scour is eliminated.

(2) The normal loss of strength of yarn in the dyeing process will be reduced.

(3) The presence of oil will assist weaving and knitting.

(4) The extent of foaming, which is a serious problem with certain dyes, will be reduced.

(5) The general levelness of dyeing will be improved.

During the course of the laboratory work it has been shown that the rate of dyeing and rate of dye migration are increased when fatty acids are present on the wool. On the other hand, mineral oil and wool grease retard the dyeing rate and, since these compounds might be unevenly distributed on the wool after scouring, they could lead to uneven dyeings. It is essential that the oil contains an appreciable proportion of free fatty acid, and it has been found that the commercially available wool-processing oils containing not less than 30% free fatty acid give level dyeings with the normal range of dyes used on woollen yarns. The presence of dirt picked up during normal processing has not been found to affect levelness.

The fastness to scouring, alkaline milling, rubbing, and light of the yarns dyed in oil was similar to that obtained when the same yarns were dyed after scouring. The presence of oil on fibres other than wool reduces their dyeing rates and limits dye migration, but yarn containing 5% of nylon has been dyed satisfactorily in oil; further experiments on blended yarns are to be carried out.

A number of bulk dyeings have already been made with promising results using neutral premetallised, milling acid, acid, afterchrome, and metachrome dyes in depths up to a 16% chrome black.

G. A. SMITH

WOOL INDUSTRIES RESEARCH ASSOCIATION
BRANCH LABORATORY
GALASHIELS
21st July 1961

Nomenclature for Man-made Fibres

In connection with the comment and proposals made recently by à Brassard[1] concerning fibre terminology, it is useful to consider more closely the range of fibres for which adequate terms are required, and the American generic names and German polymer symbols which attempt to provide systematic nomenclatures for end-product labelling and technological use, respectively.

TABLE I

Fibres from Natural Polymers

	U.S. Federal Trade Commission Identification Act 1958		Trade Names (examples)	Example of main repeating unit
	Generic Name	Definition (abbreviated)		
REGENERATED CELLULOSE	Rayon	regenerated cellulose with not less than 85% OH groups unsubstituted	Fibro, Cuprama	CH_2OH ...
Cellulose esters	Acetate	cellulose acetate	Celanese, Dicel, Fibroceta	CH_3OAc ...
	Triacetate	cellulose acetate with not less than 92% OH groups acetylated	Arnel, Tricel	... $CH-O-$ (fully acetylated) AcO OAc
REGENERATED ALGINATE	—	(regenerated alginate)	Calcium alginate	$COO-Ca_{\frac{1}{2}}$ ...
REGENERATED PROTEIN	Azlon	regenerated protein	Fibrolane	$-NH-CH-CO-$ R
RUBBER	Rubber	natural (or synthetic) rubber	Lactron	$-CH_2-C=CH-CH_2-$ (natural) CH_3

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TABLE II
Fibres from Synthetic Polymers

U.S. Federal Trade Commission Identification Act 1958		Trade Names (examples)	Example of main repeating unit
Generic name	**Definition (abbreviated)**		
POLYAMIDE Nylon	polyamide	Bri-Nylon	$-NH(CH_2)_6NH-CO(CH_2)_4CO-$ (nylon 6.6)
		Brulon 255	$-NH(CH_2)_6NH-CO(CH_2)_8CO-$ (nylon 6.10)
		Grilon, Perlon	$-NH(CH_2)_5CO-$ (nylon 6)
		Rilsan	$-NH(CH_2)_{10}CO-$ (nylon 11)
POLYURETHANE Spandex	not less than 85% segmented polyurethane	Lycra, Vyrene	$-R^1-NHCOO-R^2-$
POLYESTER Polyester	not less than 85% ester of a dihydric alcohol and tere-phthalic acid	Dacron, Fortrel, Kodel, Terylene, Vycron	

$$-ORO-CO-\bigcirc-CO-$$

$$(-OCH_2CH_2O-CO-\bigcirc-CO-)$$
(Terylene)

POLYVINYL (TYPE)			
Cyano Derivative Acrylic	not less than 85% acrylonitrile	Acrilan, Courtelle, Creslan, Orlon, Zefran	$-CH_2-CH-$ CN
Modacrylic	35–85% acrylonitrile	Dynel	$-CH_2-CH-...-CH_2-CH-$ CN Cl
		Verel	$-CH_2-CH-$ CN
Nytril	not less than 85% vinylidene dinitrile	Darvan	CN $-CH_2-C-$ CN
Chloro Derivative			
Vinyon	not less than 85% vinyl chloride	Rhovyl	$-CH_2-CH-$ Cl
		Vinyon	$-CH_2-CH-....CH_2-CH-$ (90:10) Cl $OCO\cdot CH_3$
Saran	not less than 80% vinylidene chloride	Saran, Tygan	Cl $-CH_2-C-$ Cl
Hydroxy Derivative Vinal	not less than 50% vinyl alcohol, and not less than 85% vinyl alcohol plus acetal units	Vinylon	$-CH_2-CH-$ (with methylene bridges) OH
POLYOLEFIN Olefin	not less than 85% ethylene, propyl-ene or other olefin	Courlene, Courlene X3	$-CH_2-CH_2-$
		Courlene PY	$-CH_2-CH-$ CH_3
Substituted —	(polystyrene)	Polyfiber	$-CH_2-CH-$ C_6H_5
—	(polytetrafluoro-ethylene)	Teflon	$-CF_2-CF_2-$
GLASS Glass	glass	Fibreglass	O $-O-Si-O-$ (and metal ions) O
METALLIC Metallic	metal, plastic-coated metal, metal-coated plastic, or metal covered	Lurex	—

Tables I and II classify the man-made fibres (a term which, however clumsy, is likely to be used until a generally acceptable alternative is found) according to derivation from natural or synthetic polymers. The sub-headings indicate different chemical types, and within the sub-headings are listed different chemical identities. The tables include most fibres of commercial importance in the western world. Where available, the United States Federal Trade Commission's generic names and definitions are shown. Examples of American, British, and other trade names are given, although the authority of the generic names extends only to fibres used within the United States.

TABLE III

Symbols— DIN 60002 (supplemented by Koch)

PA	polyamide
PE	polyester
PF	polytetrafluoroethylene
PT	polyethylene
PVA	polyvinyl alcohol
PVB	polystyrene
PVC	polyvinyl chloride
PVD	polyvinylidene chloride
PVM	copolymers (e.g. PVC-PVY, Dynel; PVC-PVD-PVY, PeCe 120)
PVN	polyvinylidene cyanide (dinitrile)
PVY	polyacrylonitrile
PU	polyurethane
S	filament yarn
F	staple fibre
R	monofilament
fR	fine monofilament
H	bristle
B	strip
T	tow
+	spun-dyed

In Germany the specification DIN 60002 listed recommended abbreviations for synthetic polymers which, with some more recent additions by Koch [2], are given in Table III. The specification also gives symbols for fibre types, and some of these are included in the table. The synthetic-polymer fibres referred to in Table III are usually termed "Synthetische Fasern", while fibres manufactured from natural polymers are frequently described as "Fasern aus natürlichen Rohstoffen", although the alternative "Halbsynthetische Fasern" has been used. The usual German term corresponding to man-made fibres is "Chemische Fasern" or its contraction "Chemiefasern". It is of interest that in Germany "Reyon" refers only to viscose-process fibres, cuprammonium-process fibres being referred to as "Cupro", or by the terms "Chemiekupferseide" (filament yarn) or "Kupferspinnfaser" (staple fibre).

The purpose of this note is not to make new proposals, but to survey the wide range of materials that require identification by a suitable nomenclature, and to show the progress made in this respect elsewhere. Ultimately, with the production in Britain of an increasing number of fibres, and with the prospect of wider trade, agreed generic terms will become highly desirable in order to avoid the confusion inevitable from a large number of fibre trade names, or from the haphazard use of abbreviated terms.

J. E. FORD

COTTON SILK AND MAN-MADE FIBRES
    RESEARCH ASSOCIATION
SHIRLEY INSTITUTE
DIDSBURY
MANCHESTER 20
13th July 1961

[1] à Brassard, J.S.D.C., **77**, 210 (1961).
[2] Koch, *Z. ges. Textilind.*, **60**, 17 (1958)

Man-made Fibres

In the context of this correspondence are there not just natural and unnatural fibres: or is this too simple for the technological garble of our time?

G. S. MARR

BRITISH COTTON AND WOOL DYERS' ASSOCIATION LTD.
WOOLFOLD
BURY, LANCASHIRE
28th July 1961

(*By a man-made decision, this correspondence is now closed— Editor*)

Notes

Election of Associates

At the meeting of Council on 6th September 1961 the following, having passed the prescribed examination, were elected Associates of the Society—

Peter David Bailey

Nottingham; Dyeing Technologist, Chemstrand Ltd

Anthony Peter Biddulph

Nottingham; Laboratory Assistant, H. Ashwell & Co. Ltd

William Allan Blue

Dunblane, Perthshire; Works Chemist and Assistant Colourist, British Silk Dyeing Co. Ltd

Richard Guy Charlesworth

Glenfield, Leics.; Dyer, A. E. Charlesworth & Co. Ltd, Leicester

Stanley Tordoff Drake

Fixby, Huddersfield; Dyeing Technologist, Yorkshire Dyeware & Chemical Co. Ltd, Leeds

George Terence Dyson

Halifax; Apprentice Dyer, Bradford Dyers' Association Ltd, Bradford

Douglas Walker Ford

Wakefield; Textile Chemist, Harrap Bros. (Sirdar Wools) Ltd

William McClughan Hall

Liverpool; Assistant Chemist, Johnson Bros. (Dyers) Ltd

Michael James Hamilton

Bradford, Yorks.; Chemist, Allied Colloids Manufacturing Co. Ltd

Brian Hobson

Clackmannan; Dyer, Patons & Baldwins Ltd, Alloa

David William Jones

Nottingham; Chemist, Cox, Moore & Co., Long Eaton

James McCartney Jnr.

Newtownards, N. Ireland; Works Chemist, Ulster Print Works

George John Murfet

Nottingham; Laboratory Assistant, Lindley & Lindley Co.

Robert Anthony Nickson

Crumpsall, Manchester; Assistant Technical Officer, Imperial Chemical Industries Ltd, Blackley

Matthew Joseph Olley

Claybrooke Magna, near Rugby; Assistant Chemist, Sketchley Ltd, Hinckley

Neil John Plumb

Halstead, Essex; Assistant to Head Technologist, Courtaulds Ltd, Bocking

Frank Porter

Prestwich; Technical Laboratory Assistant, Geigy Co. Ltd, Middleton

Brian Rostron

Prestwich; Works Chemist, J. H. Bleackley Ltd, Manchester

Bernard Saunders

Rochdale; Chief Chemist, Samuel Heap & Son Ltd

John Maxwell Scrimshaw

Nottingham; Dyer and Chemist, W. E. Saxby Co. Ltd

John Shore

High Lane, near Stockport; Chemist, Calico Printers' Association Ltd, Manchester

David Thomas

Nottingham; Foreman Dyer, Weldon & Wilkinson

Council Meeting— 5th July 1961

Among the matters discussed at the meeting of Council on 5th July were—

PRESENTATION OF MEDALS AND DIPLOMAS— On the recommendation of Diplomas Committee it was agreed that a ceremony for the presentation of Medals and Diplomas similar to that which took place in Bradford in March of this year should be held in Bradford in 1962.

MEMBERSHIP— 27 Ordinary Members and 6 Junior Members were elected.

REPRESENTATION ON OTHER BODIES— Mr. R. J. Hannay was appointed to represent the Society on the Advisory Subcommittee for Chemistry of the Leeds College of Technology.

Presentation to *Journal* Indexer

Mrs. E. G. Thomson recently resigned as *Journal* Indexer, a post that she had occupied for the last ten years. At the July meeting of Council unanimous agreement was given to a resolution of the Finance and General Purposes Committee that a suitable gift should be made to Mrs. Thomson in recognition of her long and faithful service in indexing the *Journal*. The gift, a suitably inscribed gold watch, was recently presented to Mrs. Thomson by the President of the Society, Mr. R. J. Hannay.

Annual Golf Competition for the Goodall Trophy

This year, for the first time, the competition was linked with the various area golf meetings. The

change appears to have been generally acceptable and resulted in a greatly increased entry of more than 80 and it is proposed to continue with the same procedure in future years. It is hoped that more areas will be able to arrange golf meetings for May 1962. The winner was Mr J. S. Falconer, competing in the Scottish Section meeting at Callander on May 27th, who returned a score of $82 - 17 = 65$ net.

Shirley Institute Honour

At a recent luncheon of the Council of the Cotton, Silk, and Man-made Fibres Research Association, Mr. N. G. McCulloch, C.B.E., was appointed to the Association's Board of Honorary Life Governors in recognition of his outstanding services to the Association and the industry. Mr. McCulloch, who is a Fellow of the Society, has been associated with the Shirley Institute for over 20 years and was Chairman from 1949 to 1960.

British Colour Council

Sir Ernest W. Goodale was recently re-elected President of the Council and Mr. Henry G. Dowling re-elected Chairman.

American Association of Textile Chemists and Colorists

Since its foundation in 1921 the A.A.T.C.C. has had its headquarters at the Lowell Technological Institute, Lowell, Mass. Because of the need for space for the expansion of the Institute and the desire on the part of the Association to establish its national office in a central location, a decision has recently been taken to move the headquarters to New York City. It is believed that this will have advantages for members, since a large segment of the membership is found in the middle Atlantic area; other advantages cited include its proximity to the Association's publishers and the desirability for a metropolitan location with a concentration of technical society headquarters of similar organisations.

Textile Institute Finishing Group

The third conference of this Group will be held in the Lesser Free Trade Hall, Manchester, on Wednesday, 25th October 1961, on the subject of "Mechanical Finishing". Full details are published in the August issue of the *Proceedings* section of *J. Textile Inst.*

O.C.C.A. Fourteenth Technical Exhibition

The 14th Technical Exhibition of the Oil and Colour Chemists' Association will take place from 26th February to 1st March 1962 in the Old and New Halls of the Royal Horticultural Society, Westminster, London, S.W.1. The theme will be the presentation of technical advances in those industries supplying the paint, varnish, printing ink, linoleum, and allied industries.

Chemical Society Research Fund

The Research Fund of the Chemical Society provides grants for the assistance of research in all branches of chemistry. Applications for grants will be considered in November next and should be submitted on the appropriate form not later than Wednesday, 15th November 1961. Applications from Fellows of the Chemical Society will receive prior consideration. Forms of application, together with the regulations governing the award of grants, may be obtained from the General Secretary, The Chemical Society, Burlington House, Piccadilly, London, W.1.

Symposium on Carbohydrate Chemistry

An international Symposium on Carbohydrate Chemistry, sponsored by the Chemical Society, in association with the University of Birmingham, will be held in Birmingham during the period 10th–20th July 1962. Further particulars will be published in due course. Copies of the announcement will be sent, when available, to those who apply to the General Secretary, The Chemical Society, Burlington House, London, W.1. Papers read at this meeting will not be published in full in collected form.

Mechanism of Dye Formation by Photo-reactions of Triarylmethyl Leuconitriles

Triarylmethyl leuconitriles can be used as photographic materials which produce a visible image immediately on irradiation without subsequent development. Thus Malachite Green leuconitrile can form the dye when exposed to ultraviolet radiation, depending upon the dielectric constant of the solvent present. If the solvent has dielectric constant > 4.5, e.g. ethanol, dye formation readily occurs, but in cyclohexane and other solvents of low dielectric constant, irradiation produces a photoreaction but no dye. A. H. Sporer (International Business Machines, San José, California) now states that there are two photoreactions. One, occurring only in solvents of high dielectric, involves hydrolytic cleavage of the nitrile group and leads to the dye— a triarylmethyl cation— via a triarylmethane free radical. The other, which occurs in all solvents, involves homolytic cleavage of substituents on the amine nitrogen. The quantum yield of the dye-forming reaction had previously been reported as essentially unity. The cleavage reaction was entirely overlooked because its quantum yield is only 0·02; this is within the experimental error of the determinations on the dye-forming reaction. Yet another type of reaction occurs with Victoria Blue B leuconitrile. When this compound is dissolved in ethanol and irradiated at room temperature, intense fluorescence results, but no dye is formed. In this case about half the radiation is absorbed by the dimethylaniline groups. The other half is absorbed by the N-phenyl-1-naphthylamine group and produces fluorescence. But the radiation absorbed by the dimethylaniline groups produces neither cleavage nor dye-forming ionisation. Instead this energy is transferred to the N-phenyl-1-naphthylamine group and it too is emitted as fluorescence (*Chem. Eng. News*, **39** (21) (22 May 1961) 42). C.O.C.

U.S.A. Production and Imports of Dyes in 1960

According to the U.S. Tariff Commission, U.S.A. production of dyes in 1960 was 156 million lb, being 8% lower than in 1959, but 11% higher than in 1958. Sales of dyes were 148 million lb, value $192 million, compared with 159 million lb, value $206, in 1959. Of the total production of dyes, 22% were azo, 22% anthraquinone, 20% sulphur, 6% indigoid, and 6% stilbene. Compared with 1959 there was a drop in production of 19% in indigoid, 15% in azo, 6% in stilbene, 2% in sulphur, and 1% in anthraquinone dyes. Imports of dyes (excluding organic pigments) were valued at $7·6 million in 1960 and $7·9 million in 1959, a decrease of 4%.

C.O.C.

Meetings of Council and Committees
August

Conference Subcommittee of International Relations— 11th

Publications— 22nd

Colour Index Editorial Board— 28th

New Books and Publications

Organic Coating Technology Volume II
Pigments and Pigmented Coatings

By H. F. Payne. Pp. viii + 725. New York and London: John Wiley & Sons 1961. Price, 140s. 0d.

This is a comprehensive yet concise book about paint making, and is a companion to Volume I, *Oils, Resins, Varnishes and Polymers*, by the same author. It is an extremely sound blend of fundamental principles and practical information, and there is throughout a commonsense approach displaying a wide knowledge of the industry and its detailed problems.

Besides being useful to the beginner in the paint industry, it should prove of value to the experienced paint maker as a concise statement of his fundamental concepts and general and detailed principles of formulation. There is a certain amount of intentional repetition from one section to another, but this makes each section more self-contained. In addition there are frequent cross-references which enable the complete picture on any one aspect to be readily formed. Furthermore, sound principles are always worth establishing beyond doubt. The subject has not been over-simplified and no important points appear to have been overlooked. For the more advanced student or experienced paint maker there are frequent and ample references to the literature.

It should, however, be noted that the book is primarily concerned with American practice, and the vast majority of the references relate to American journals, the remainder being mostly British journals. In addition, the author refers extensively to raw materials by their trade names, a practice which is not usual in this country but which in a subject of this type has considerable advantages. Thus, although general information and general formulations can be given without reference to trade names there is often a significant variation between manufactured products which are ostensibly the same, and manufactured raw materials are often designed with undisclosed know-how in order to suit them for specific applications. It is therefore useful to have formulations including named products. This does not limit the value of the book because fundamental principles are kept well to the fore.

The book is well balanced between descriptions of white, extender, coloured inorganic and organic, black, and metal pigments and metallic stearates on the one hand and fundamental principles, methods of preparation and application, and different types of paint on the other.

Coloured pigments are well covered, although modern colorant makers will be surprised to learn that large wooden vats are still used. The amount of space devoted to Lithol Reds, Lake Red C, Persian Orange and Tartrazine lakes may be justified for the American market, but these pigments are not used extensively in paints in the United Kingdom. Phthalocyanine Blue is adequately discussed and the reference to the hazing of Toluidine Red illustrates the good coverage of small but important practical details. The quinacridone red and violet pigments are described, illustrating the fact that the book is well up to date.

The style is good and the British reader will find the terminology acceptable. Technical expressions used in the industry are well defined, and confusion in this connection often experienced within the industry is avoided. Testing methods are adequately described, but for fuller details reference is always made to the A.S.T.M. designations. One must sympathise with the author when he regrets that it is impossible to publish a book of this type which is completely up to date. He has succeeded extremely well and one can merely note that there is no reference to the plate and cone viscometer or to the Xenotest light-fading outfit.

It is gratifying to note that periodic revision of the two volumes has been arranged. This is particularly important in a book of this character, but it should nevertheless be stressed that fundamental principles and the commonsense approach are of lasting value. Thus the book, although particularly recommended for the student, will be of great interest to all connected with the paint industry and the use of pigments at any level.

F. M. SMITH

Non-convertible Coatings

Collator: J. B. G. Lewin. Pp. 326. London: Chapman & Hall (on behalf of Oil and Colour Chemists' Assocn.). 1961. Price, 30s. 0d.

This book is Part I of a work on paint technology to be published in six parts, and the Foreword

states that it follows closely the syllabus for the theoretical and practical work of the Non-convertible Coatings Section of the City and Guilds Examination in Paint Technology. This reviewer is not familiar with that syllabus but can readily imagine that this statement is correct, for the book appears to omit nothing of any importance relative to the materials covered by its title. It is an excellent production and it is certain that the hopes expressed in the Foreword will be realised, viz., that "although the primary purpose of this manual is to help students preparing for examinations, it is hoped that it will also assist not only graduates who have newly entered the coatings industry, but their more experienced seniors as well".

The book deals lucidly not only with such matters as the theoretical aspects of the nitration of cellulose, the mechanism of plasticisation, and the mechanism of polymerisation, to mention three items picked at random, but also provides a large number of detailed formulations for finishes of various kinds. It is obvious that the ten authors and the collator are practical technologists and chemists who know their subjects intimately and at first hand; as a consequence, very few factual errors were detected. This is not to say that the book is perfect. It is not; it suffers, as this type of book usually does, chiefly from the fact that it is the work of a number of authors, leading to a certain unevenness of treatment and particularly to a degree of repetition and inconsistency of nomenclature. Since this is a text book, as well as a work of reference, this is of some importance.

A non-convertible coating is briefly defined as one which does not undergo any significant chemical change on continued exposure. It is not surprising to find, therefore, that nitrocellulose lacquers occupy approximately half the book and are allotted six chapters, the first being devoted to the manufacture, types, testing, properties, etc. of nitrocellulose itself, while the next three chapters deal with solvents for nitrocellulose lacquers, plasticisers, and pigments suitable for lacquers. Chapter 5 discusses the formulation of wood finishes and Chapter 6 principal types of lacquer— a rather strange order. The chapter on plasticisers also deals with resins, general principles of formulation, and the manufacture of lacquers. The very detailed chapter on wood finishes contains a short section in which convertible coatings of the polyester type are described, and in spite of the disclaimer in the Foreword the reviewer thinks this section could well have been left for Part III, where such finishes could have had the much fuller treatment they merit.

Chapter 7 covers cellulose acetate and includes cellulose acetate butyrate, and Chapter 8 is on ethylcellulose. Chapter 9 is concerned with the chemical derivatives of rubber, including chlorinated rubber, cyclised rubber, and oxidised and depolymerised rubber. An excellent chapter follows on synthetic thermoplastic lacquer resins, which covers polyvinyl chloride, vinyl chloride copolymers, copolymers of polyvinylidene chloride, polyvinyl acetate, alcohols and acetals, acrylic and methacrylic ester polymers, polystyrene, coumarone and indene polymers, and vinyl ether polymers. The final chapter deals with spirit varnishes, including shellac, other natural resins, and spirit-soluble phenolic resins. Every chapter concludes with a number of useful practical experiments.

Attention may be drawn to a few items: safety goggles should be worn when handling dry nitrocellulose. Among the important uses of nitrocellulose and of polyvinylidene chloride–acrylonitrile copolymers that might have been mentioned is coating of transparent viscose film for heat-sealing and moistureproofing. Some confusion regarding lacquers that may be described as "high-flash" and the object and interpretation of the Cellulose Solutions Regulations is evidenced on pp. 58, 192, and 271. On p. 132 Cellolyn 501 is described incorrectly as a hard maleic resin; it is in fact a non-drying oil alkyd. The solution of DHL 25/45 in butyl acetate in the formulation on p. 149 would be so thick as to be unmanageable. There are several spelling mistakes, e.g. Arochlor for Aroclor, and the captions to the photographs on p. 221 are in fairly obvious conflict with the text on p. 220, while on p. 278, reference to Fig. 3 should presumably be to Fig. 26, which is not reproduced at all; the same applies to Fig. 4 on p. 279. The asterisk in Table 31 has no apparent significance.

These errors will be recognised as of minor importance in a book which is excellently printed and produced. At the very reasonable price it should be in the hands of all surface-coatings technologists.   J. CREASY

Printing Ink Manual

Editor-in-chief: R. F. Bowles. Pp. viii + 746. Cambridge: W. Heffer & Sons Ltd. (for Society of British Printing Ink Manufacturers). 1961. Price, 70s. 0d.

This book, which was commissioned by the Technical Training Board of the Society of British Printing Ink Manufacturers, has collected together authoritative and up-to-date information for the help and guidance of all those connected with the printing ink industry. But even more particularly this book is intended to help students to meet the rising standard necessary to pass the City and Guilds examinations in Printing Ink and Roller Technology.

The contents of the volume are divided into two parts. The first gives brief historical details followed by an account of printing processes. Information is given about the nature of the surfaces which are printed upon, and there is also a section dealing with colour and colour vision. Part II consists of a detailed account of the nature, behaviour, and manufacture of printing inks in relation to the various printing processes. This section also contains a chapter dealing with print recognition and printing defects.

Because of the diversity of the subject and the detail in which it is treated, a book has been produced which is rather bulky and difficult to handle. It is also expensive, particularly as it is intended for students. Furthermore, technical

though the book may be, the layout is, perhaps, somewhat dull and unimaginative. The photographs sometimes serve no instructive purpose and some seem old-fashioned. Surely in a book produced by and for an industry whose existence depends upon colour one might have had some lively use of colour itself, but perhaps this would have further increased the price of the book.

Twenty-two scientists and technologists have contributed to this book and consequently the way the material is presented varies considerably. Much is written in disorganised note form; some information is not sufficiently detailed, whilst some is too condensed. There are many instances of repetition of fact. For example, information concerning printing processes in Part I is repeated in Part II. The impression of haste in one or two sections does not always inspire confidence. The chapter on colour and colour vision and that on paper, excellent though they are, are disproportionately long and upset the balance of the book.

Nevertheless, this is a formidable book. It is the only one of its kind that makes readily accessible factual information, some of which is being published for the first time. Many sections are eminently readable, particularly the chapters giving an account of the individual types of inks. Here a most successful attempt has been made to correlate ink and its press behaviour, and one soon has an impression that this has been achieved by sound ink knowledge obtained by practical experience.

When the book is reprinted, the inaccuracies must be corrected. For instance, on page 141 the definition of MSAT film is that of MSADT film. Page 331 gives the relative evaporating time of isopropyl acetate as 42 instead of 4·2. Also, it would be desirable in a reprint to devote some space to simple spot tests for resins and pigments, since students spend much of their time identifying pigments when colour matching. These minor criticisms apart, the Printing Ink Manual with its wealth of information is a most useful book. As a comprehensive reference and guide to all those connected with the industry it has no equal.　　　　R. J. PIERCE

250 Years of Clothworking in London

London: Perrotts Ltd. 1960. No price.

In a booklet issued by Perrotts to mark the 250th anniversary of the entry of a Perrott into the wool trade in 1710, the history of woollen goods is traced in Britain over the last 3000 years. British cloth was mentioned in the Edict of Diocletian in the 3rd century A.D. and again in the first recorded commercial treaty in the 8th century. After the Norman conquest the craft began to expand rapidly and "gilds" of weavers were established. Trade expanded and by the middle of the 14th century our export trade amounted to 5000 cloths (each 24 yards long by 2 yards wide).

As the industry grew, some degree of specialisation developed and carders, spinners, weavers, and finishers emerged— the last-named again specialising into scourers, fullers (the first mention of a fulling mill being to that at Calverley in 1175), raisers, shearers, and so on. Later the term "finishing"

grew to mean the processes subsequent to dyeing, including the famous London Finish, and, in comparatively modern times, permanent creasing, and shower-, moth- and fire-proofing.

The booklet, well written and produced, contains a wealth of information presented in an interesting manner. It will be read with pleasure by historians and all engaged in the wool trade.　　A. THOMSON

The Dyeing of Cellulosic Fibres and Related Processes

By S. R. Cockett and K. A. Hilton. Pp. xiii + 417 with 64 fig. and 30 plates in the text. London: Leonard Hill (Books) Ltd. 1961. Price, 50s. 0d.

It may be said at the outset that this book contains a great deal of information and its price is reasonable. It is even wider in scope than the title suggests for, in addition to the 99 pages devoted to structure and processing and 23 to finishing, there is an 88-page section on the chemistry of colouring matters, one of 39 pages on theory of dyeing, and one of 33 pages on light and colour in relation to chemical constitution. The colouring of cotton textiles is confined to considerably less than half the book.

The inclusion of the chapter on colour and constitution is really out of place in a text book on cotton dyeing, but could be justified on the grounds that the information is not readily available elsewhere. The present treatment is too brief to be really adequate. Not enough stress is laid on π-orbitals, and molar extinction coefficient (used in the graphs) is not defined.

The account of dyeing theory is a praiseworthy attempt to deal with a difficult subject. It might have been better to give less space to affinity and to have included "time of half-dyeing" and "activation energy". Little attempt has been made to assess the significance of theoretical studies of dyeing.

The chemistry of colouring matters is dealt with on the basis of a scheme of classification by chemical constitution, but not strictly according to the characteristic chromogen present. The result is that there are separate sections for azo, anthraquinone, xanthene, etc., compounds and also for reactive dyes and onium dyes, where the emphasis is on the grouping primarily responsible for dyeing behaviour. It might have been better if the relevant colour chemistry had been incorporated as an introduction to the application of each main class of dye. Too much space is devoted to dyes of little or no importance on cellulose, e.g. acid wool dyes and basic dyes, while the fast-to-light direct dyes based on cyanuric chloride and a complex oxazine structure are not mentioned. There are errors, some hard to excuse, e.g. that in which alizarin is credited with containing a salicylic acid residue (p. 147). Incidentally, though reference is made to the 2nd Edition of the *Colour Index*, C.I. numbers are not given.

The application of dyes is covered under four heads, viz. directly and indirectly substantive dyes; solubility-cycle dyes; colouring matters formed *in situ*; and resin-bonded pigments. More

emphasis on the fundamental objectives of the colourist and the significance of the various stages in coloration would have helped the student to grasp more easily the essentials of each process and would have encouraged him to form objective judgments.

Although the book covers so wide a field there is no section dealing with the specification and determination of colour fastness. Viscose rayon does not receive much attention. References are given to a number of text books, but there is no reference to monographs on such topics as continuous dyeing which, though published by dye makers, are widely available and of considerable value. The almost total omission of references to papers in scientific journals is difficult to defend, especially since sundry authors are mentioned by name, and the Society's particular contribution through the explanatory papers on modern theory could well have kept company with the solitary reference on p. 249.

There are numerous useful photographs of dyeing machinery and other equipment, as well as diagrams, some of which would be improved by having additional captions. The variety of sizes in which formulae are presented, even on the same page, is irritating, as are also various minor errors and some confusion in setting out, but this is, presumably, the fault of the printer.

This is the first book, for a long time, which has attempted to deal comprehensively with the coloration of a particular type of fibre. The deterrents facing any author are as considerable as the difficulties are formidable, and the present authors deserve our thanks for providing a much-needed text book which, despite its faults, will be of considerable value to students of dyeing.

C. B. STEVENS

Dictionary of Dyeing and Textile Printing

By H. Blackshaw and R. Brightman. Pp. vi + 221. London: George Newnes Ltd. 1961. Price, 30s. 0d.

In any society, accurate communication of facts, ideas, and opinions amongst its members is greatly facilitated when a vocabulary of well-defined terms is available. This Society, in its *Journal* and the parlance of its members, has inherited as its basic vocabulary the traditional terms of the textile dyeing, printing, and finishing industries, which are often ill-defined and subject to local variation. To this basic vocabulary new terms are continually being added in relation to new products or processes or through efforts to describe and explain the phenomena of dyeing and finishing in a more scientific manner.

The Society's Terms and Definitions Committee, formed 10 years ago, set out to define such terms as appeared to warrant it, and this has proved a time-consuming task. After much anxious and careful deliberation, the Committee has given its blessing to some 160 definitions, comprising traditional, modern, and scientific terms. This glossary is rather inaccessible, being published only in the *Journal*, but many of the definitions have been adopted by the corresponding Textile

Institute committee and incorporated in its publication *Textile Terms and Definitions*. The fourth edition of this authoritative work contains about 1600 definitions, the majority of which are, however, concerned with the manufacturing sections of the textile industry.

The present dictionary must be welcomed as providing under one cover a collection of some 1500 terms, relating specifically to the wet-processing side of the industry, and prepared as a private venture by two experienced former members of the staff of I.C.I. Dyestuffs Division. The majority are technical terms mainly concerned with the chemicals, dyes, and other raw materials of the industry, and their uses and methods of application, which are dealt with most comprehensively. Scientific and commercial terms are also included where appropriate, and two Appendices comprise a short bibliography of dyeing, printing, and finishing literature and an index of trade-marks of commercial man-made fibres; inevitably the latter is somewhat out of date.

The text is well laid out, with clear typography, on good paper, which should withstand much thumbing, as befits a work of reference.

The definitions are usually short and to the point, but are in many cases clarified further by explanation and example and by the extensive use of cross-references; this will undoubtedly enchance the value of the dictionary, particularly to the student. In several cases, the definitions of machines are accompanied by diagrams and, apart from the last one, which shows a most unrealistic winch-dyeing machine, these are clear and informative. The authors have, with the Society's permission, adopted most of the definitions published in the *Journal* and thus make them more accessible to the general reader. It is understood that it was not possible to draw on material from *Textile Terms and Definitions*, although a number of relevant terms have been defined.

It is almost inevitable that in a first edition of this type of work there will be errors, but the authors are to be congratulated in this respect. Typographical errors are almost negligible and the number of factual errors has been kept very small, particularly in respect of the technical terms defined. Those errors which have crept in are usually concerned with the more chemical or physical aspects of definition; there are also a few important omissions in this field.

Although this new dictionary cannot yet command the status of the Textile Institute publication, it is a most useful complementary work of reference for authors, librarians, editors, and other specialists; it should also go far towards meeting the needs of students and other non-specialists coming into this field.   J. S. WARD

Polypropylene

By T. O. J. Kresser. Pp. xi + 268. New York: Reinhold Publishing Corpn. 1960. Price, 52s. 0d.

This volume is the latest in the Reinhold Plastics Applications Series and, like its predecessors, provides a semi-technical treatment of

interest to those concerned with the fabrication and marketing of plastics.

The excitement generated in recent years by the development of stereoregular polymers and, in particular, by polypropylene, may tempt many readers to this book, but those outside the field of plastics applications are likely to be disappointed. Both the polymer scientist and the plastics technologist will find no more than a general introduction to their particular interests: the value of this volume is severely restricted by the almost complete absence of references to the original literature. For the reader with purely textile interests, Kresser's book is a special disappointment which may be exemplified in the brief treatment of polypropylene fibres: the suggestion is made that certain yarns may be produced by wet or dry spinning rather than by melt spinning, but the account ends at this tantalising stage. Some discussion is given of the dispersion of pigments in polypropylene melts but none on the dyeing of fibres.

The final four chapters of this volume are largely devoted to predictions of the nature and extent of future markets for this polymer. The book is well produced and appears free from misprints; the line drawings are admirably clear. As in so much of plastics literature, the text is broken by an occasional photograph whose only virtue is a mild decorative charm.   G. J. HOWARD

Telomerisation and New Synthetic Materials

By R. Kh. Friedlina and Sh. A. Karapetyan. Translation edited by B. P. Mullins. Pp. x + 102. Oxford: Pergamon Press. 1961. Price, 25s. 0d.

Telomers, or low-molecular-weight polymers, have important uses as intermediates in the production of fibres, plastics, and other materials. Their production may involve long and uneconomic routes but they may be economically produced by the so-called "telomerisation reaction"—

$$XY + n\, CR_1R_2{=}CR_3R_4 \rightarrow X(CR_1R_2CR_3R_4)_n Y$$
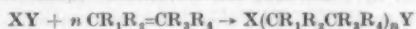
This book is concerned with telomerisation generally and more particularly with the production of tetrachloroalkanes and their conversion to substances of industrial importance. The production of the fibre "enanth" from amino-enanthic acid is described and its properties are compared with those of other fibres. The book is intended for readers with an average school knowledge of the fundamentals of chemistry and physics and, because of this, it includes much material likely to be familiar to readers of the *Journal*.

The first chapter outlines the concept of macromolecules, polymers, polymerisation, and the telomerisation reaction. The second is concerned with the technology of telomerisation and considers the reaction between ethylene and carbon tetrachloride at a temperature of 100°C and pressures below 150 atmospheres. The effects of variations in concentration of reactants, temperature, pressure, and time are discussed, and methods of separating tetrachloroalkanes are outlined. A short third chapter deals with the conversion of telomers and outlines methods by which terminal chlorine atoms of tetrachloroalkanes can be replaced by functional groups capable of reaction to give fibres, plastics, and other substances.

A longer fourth chapter is concerned with the "enanth" fibre and describes the conversion of tetrachloroheptane to amino-enanthic acid, the polymerisation of the acid, and the extrusion of the polymer to form fibres. Elementary accounts of synthetic-fibre formation, fibre-forming polymers, and the strength of fibres are included, and a comparison is made of some properties of "enanth" fibres with those of some other fibres. The final chapter deals with other materials obtained from tetrachloroalkanes, including a fibre-forming copolymer of amino-enanthic acid and caprolactam, plastics, plasticisers, and perfumes.

The enthusiasm of the authors for telomerisation sometimes leads to statements with which others may not agree. Thus, whether the pressures involved in the production of high-pressure polyethylene constitute a big enough obstacle to limit its industrial production is perhaps debatable. Russian contributions are emphasised and the few specific references given are Russian. The production of the book by non-letterpress and photolithography does not seriously detract from its value, but some illustrations are not very clear. These are minor criticisms of a book which, within the limits imposed by the level adopted by the authors, gives a good account of the production and properties of the "enanth" fibre and the production and industrial uses of tetrachloroalkanes as intermediates. Translation appears to have been good and the price is not excessive.

                                        W. R. MOORE

Laboratory Handbook on Toxic Agents

Editor-in-Chief, C. H. Gray. Pp. viii + 170. London: The Royal Institute of Chemistry. 1960. Price, 20s. 0d.

This publication, produced by the Royal Institute of Chemistry under the guidance of its Publications Committee, provides in a condensed, authentic, and readily available form the essential information for laboratories and chemical stores on the hazards of the normal chemicals used in laboratories and in industry. Special chemicals used occasionally in research laboratories or in specialised industries are not covered, since it is assumed that such users would take more than normal precautions.

The various sections have been written by eminent authorities on the topics discussed. The first section gives a succinct account of the physiological action of toxic chemicals; the second covers general precautions and methods of preventing accidents with chemicals. The first part of the book ends with a brief section on first-aid in the laboratory.

The next section lists the main details of poisonous and corrosive gases, reagents, and solvents in alphabetical order, giving in a standard format for each chemical illustrated— (a) type of hazard, (b) acute effects, (c) chronic effects, and (d) first-aid action. This section has been compiled

in collaboration with the Medical Inspectorate of Factories. The final section of the book deals with precautions against radiation.

The main body of detailed information is made immediately identifiable by being printed on blue paper, in contrast to the white paper of the other sections. The whole book is well and stoutly bound in a distinctive red cover which will withstand normal laboratory bench uses. At the price the book is of outstanding value and should undoubtedly appear in every laboratory or works where chemicals are in constant use.

R. L. ELLIOTT

Applied Organic Chemistry

By E. Kilner and D. M. Samuel. Pp. xi + 484. London: Macdonald and Evans Press. 1960. Price, 50s. 0d.

There has always been a gap between text-books primarily designed for the teaching of pure sciences and those which deal with detailed studies of specific technologies. This gap is particularly noticeable in pure and applied organic chemistry, where most teaching books are primarily concerned with the pure science. Students therefore tend to learn the subject as a series of memorised reactions without ever recognising the significance, general or specific, of these reactions in industrial processes. This book therefore comes as a timely attempt to relate technological processes to their organic chemical background at a level well within the comprehension of the average higher-course student.

In attempting to avoid the routine classifications of academic organic reactions, the book tends to degenerate in parts into a curt list of compounds and their uses, without any explanation of the reasons for these uses. Thus thioglycollic acid is mentioned as being used for the permanent waving of hair, but no indication is given as to why this acid should be specially selected for this purpose. Again, the use of acetoacetic ester in the production of pigments is mentioned, but the reasons for its reactivity and its tautomeric nature are not given. The authors have been over-zealous in the avoidance of academic formulation, as instanced by the small number of reaction equations given, and this at times tends to detract from the value of the text. Though the authors state in the preface that the book should be used as a supplement to existing text-books of general organic chemistry, some stress on the reasons for the industrial use of specific materials from the standpoint of standard reactions of these compounds would strengthen the link with pure organic chemistry without encroaching too much into the field of the general text-book.

The book covers an enormous field of modern work and small errors or out-of-date statements are bound to occur. Thus insulin is stated to contain four amino-acid chains and to have a molecular weight of 12,000 and it is implied that this is the only protein-type material of commercial importance whose amino-acid sequence has been elucidated. Most of these errors are of minor importance, but it is surprising that there should be

no mention of the fluoro-compounds which are now increasing in importance.

The chapter on dyes and dyeing is far more closely allied to industrial use than in the normal type of organic text-book, and gives a general account of dyes, structure, class, colour, and constitution.

The volume also contains excellent readable chapters on oils, the petroleum and related industries, high polymers, and coal carbonisation, but aromatic and heterocyclic chemicals receive rather curt treatment.

The authors are to be commended on bringing the new attitude to the teaching of organic chemistry to the forefront, and lecturers will find much in this volume which will help them to avoid the arid recital of chemical properties, but the book is definitely a supplement, not a primary text in either organic chemistry or technology.

R. S. ASQUITH

Report on the Census of Production for 1958
Part 88— Textile Finishing

Pp. iv + 16. London: H.M.S.O. 1961. Price, 2s. 6d.

This report relates to establishments engaged in the bleaching, dyeing, printing, and finishing of yarns and fabrics, including the bleaching, dyeing, and dressing of lace on commission, but excludes finishing of lace and the scouring, carbonising, and dyeing of wool and hair and wool and hair tops in association with sorting, blending, and combing.

Information was obtained from all firms employing at least twenty-five persons, whereas in previous censuses details were required from firms employing eleven or more persons in productive work. For this and other reasons the figures given for 1954 are not strictly comparable with those for 1958. The details are collected in seven tables; according to Table 1, which covers the industry as a whole, the net output fell from £61·1 million in 1954 to £57·9 million in 1958, and the number employed from 87,200 to 76,000. In Table 2 the information given in Table 1 is subdivided according to fibre-type and textile form (fibre, yarn, cloth, etc.); the net output in £ million for separate sections (1954 figures in parentheses) was— cotton 33·32 (35·77), wool 7·05 (7·77), knitted fabrics 7·49 (6·45), others 6·7 (7·6).

Other tables provide an analysis by size of enterprise within the industry, details of the work done (classified according to fibre and process), and number of employees and average salaries for the week ending 25th October 1958.   M.T.

The Textile Industry in Europe
Statistical Study— 1959: Trend in 1960

Pp. 119. Paris: Organisation for European Economic Co-operation. 1960. Price, 6s. 0d.

This survey, printed in both English and French, is in two parts— (a) comments on trends in the European textile industry in 1959, and (b) statistical tables. The first part, which occupies 30 pp., includes an 8-page appendix on the reorganisation of the British cotton industry. In the statistical

tables, details of production, manpower, invest-ment, consumption, and foreign trade of each country's industry, taken as a whole, are given first; in the second set of tables, division is according

to the separate sectors of the industry (cotton, wool, man-made cellulosic fibres, man-made non-cellulosic fibres, jute). Altogether there are 68 tables.　　　　　　　　　　　　　　M.T.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified.　Abbreviations of names of firms are listed in J.S.D.C., **68**, 23 (*Jan. 1952*) *and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

PATENTS

Carbon Black (C.I. Pigment Black 6 and 7) Plant
Phillips Petroleum Co.　　　USP 2,950,179 (17 June 1957)
　Describes a valve and conduit system whereby the initial product from any of a set of furnaces producing Carbon Black can be passed into a vent conduit or into a single common Carbon-Black-gathering conduit as desired.　　　　　　　　　　　　　C.O.C.

Apparatus for the Wet Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)
Phillips Petroleum Co.　　　USP 2,949,349 (20 Mar 1957)

Dyeing Machine
Freeman, Taylor Machines　　　BP 869,043 (12 Feb 1959)
　Describes an arrangement of valves which prevents the cover of the vessel from being opened until the pressure within the vessel has been reduced to atmospheric.
　　　　　　　　　　　　　　　　　　　C.O.C.

Turning Tubular Fabric Inside-out
Pasolds　　　　　　　　　　　BP 868,738 (24 Dec 1958)

Open-width Wet Treatment of Fabrics
B. C. Morton　　　　　　　　BP 868,425 (30 Dec 1958)
　The fabric passes up and down through the tank, being guided by rollers mounted at the top and bottom of the tank. The tank also contains at each side a frame which is given a reciprocating motion. Transverse members connecting the frames extend across the tank so as to engage with the respective runs of the fabric and cause deflection of its path. This gives much more effective treatment of the fabric.　　　　　　　　C.O.C.

Control of the Relative Humidity within Dry-cleaning Apparatus
Stanford Chemical Co.　　　USP 2,949,336 (28 May 1956)

Facilitating the Positioning of a Roller in Mangles, Calenders, etc. without Dismantling the Machine
Western Board Mills　　　　BP 868,498 (4 Feb 1958)

Friction Calenders
VEB Erste Maschinenfabrik Karl-Marx-Stadt
　　　　　　　　　　　　BP 868,135 (31 Oct 1957)
　The friction roller is individually driven and in turn drives a resilient roller. The resilient roller has an auxiliary drive to retard its rate of rotation, and a self-locking gear, the driving portion of which is connected to the auxiliary drive and the driven portion of which is connected to the resilient roller.　　　　　　　　　　C.O.C.

Pin Bars for Tentering Machines
G. Möhring　　　　　BP 871,417 (Germany 30 May 1958)
　Describes a pin bar so shaped that the fabric on the pins does not come into contact with it.　　　C.O.C.

Hot Stretching and Conditioning of Thermoplastic Web and Sheet Materials
C. A. Litzler　　　　　BP 869,349-50 (28 Oct 1957)
　The web under lengthwise tension passes along a series of alternating inlets and outlets for hot air or other treating medium. The temperature of the air supplied may vary from inlet to inlet.　　　　　　　　C.O.C.

Heating or Drying Fibrous Webs
Artos Maschinenbau Dr.-Ing. Meier-Windhorst
　　　　　　　　　　BP 867,530-1 (Germany 15 Dec 1956)
　In a machine where the freely suspended web is treated by jets of hot gas directed on to both sides of the web, profiled elements, preferably having curved surfaces of which the convex sides face the web, are placed at a dis-tance from the nozzle opposite each other on opposite sides of the web so that the distance between them and the web

is not more than half the distance between the nozzle and the web. This results in the web remaining flat and also in better heat transfer.　　　　　　　　　C.O.C.

Accurate Control of the Heating of Webs and Yarns
Industrial Ovens　　　　　BP 867,593 (19 June 1958)
　The web is led into and out of a heating chamber by means of guide rolls. It passes under tension around a dancer guide roll which can move to any position between two end positions to increase or decrease the path of the web through the chamber. The position of this dancer roll is automatically controlled by the temperature which the web attains in the chamber, so that the length of web in the chamber is varied to maintain the web in the chamber at the desired temperature.
　　　　　　　　　　　　BP 867,594 (19 June 1958)
　The dancer roll is controlled manually instead of automatically.　　　　　　　　　　　C.O.C.

Guiding Textiles or the like through Fluidised Beds
Shri Ram Institute for Industrial Research
　　　　　　　　　　　　BP 867,684 (2 June 1958)
　Particles of the bed are prevented from entering the gap between a guide roller shaft and its bearings situated on the bed by directing fine jets of the fluidising medium into the gap.　　　　　　　　　　　　C.O.C.

Open Flame Dryer for Cloth, Paper, etc.
E. A. Timson　　　　　　　BP 867,314 (27 Jan 1959)
　A machine in which fluid fuel is burnt to give a flame of 600–1800°C, the flame issuing slowly from the burner so that it is carried along in contact with the material being dried.　　　　　　　　　　　C.O.C.

Raising Machine
Franz Müller Maschinenfabrik
　　　　　　　　　BP 867,333 (Germany 15 Nov 1956)
　Describes a driving mechanism which permits fine regulation of the pile and counter-pile rollers, thus enabling fine adjustment of raising.　　　C.O.C.

Paper-web Shrinking Machine
Hunt and Moscrop　　　　BP 868,391 (17 Nov 1958)
　In a machine in which the web is compressed between a deformable moving belt and a roller while the belt contracts, use of a belt having a Shore number of 65–90 results in a paper having no internal spaces or voids.
　　　　　　　　　　　　　　　　　　　C.O.C.

Continuous Impregnation of Paper and other Webs
Olin Mathieson Chemical Corpn.
　　　　　　　　　　　　BP 868,042 (21 Apr 1959)
　A container, e.g. a long tube, has a slotted upper surface and is connected to a supply of the impregnating liquor which is supplied to the container in amount and at a rate synchronised with the speed of the web as it passes over the container and in contact with the slot.
　　　　　　　　　　　　　　　　　　　C.O.C.

Impregnating Roller
Harris Transducer Corpn.　USP 2,949,384 (27 Nov 1956)
　The material passes over a roller immersed in the treating liquid. An electro-acoustic transducer means forms an integral part of the roller so that uniform radial expansion and contraction of the entire outer cylindrical surface of the roller takes place and the material conveyed by the roller into the liquid is subjected to strong acoustic excitation. This gives much better penetration of the liquid into the material.　　　　　　　C.O.C.

Coating Apparatus
J. B. Coyne　　　　　BP 867,829 (U.S.A. 11 June 1958)
　Apparatus in which the coating is applied to or smoothed on the substrate by means of endless belts.　C.O.C.

II— WATER AND EFFLUENTS

Water Treatment in the Textile Industry
H. Engelhardt   *Textil Praxis*, **16** (Feb 1961) 172–179

Describes methods of removing suspended and dissolved matter in the treatment of water for boiler feed and wet processing of textiles. Topics discussed include filtration, de-acidification (removal of free CO_2 as bicarbonates of Ca and Mg), and removal of Fe, Mn, Ca, and Mg salts. Industrial water-softening plants are shown diagrammatically.   L.A.T.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Developments in Textile Auxiliaries
B. C. M. Dorset   *Text. Manuf.*, **87** (May 1961) 195–200

The use of auxiliaries is described and discussed for reducing burnt-gas-fume fading and oxidation-fume fading (O-fading) of certain aminoanthraquinone dyes on acetate and triacetate materials; aiding the level-dyeing or stripping of unevenly dyed goods; producing heavy depths with disperse dyes on acrylic fibres; improving the wet crease-shedding power of cellulosic fabrics; improving the resistance of polyesters to photochemical decomposition; imparting crease-resist finishes to cellulosic fabrics without serious loss of strength or subsequent chlorine retention; producing dense woollen fabrics without a milling treatment; and imparting fast-to-washing antistatic finishes to synthetic fibres.   S.B.D.

Interaction of a Surfactant with a Monolayer Studied by a Radiotracer Method
R. Shimozawa and R. Matuura   *Bull. Chem. Soc. Japan*, **34** (Mar 1961) 457–458 (*in English*)

The interaction of sodium dodecyl sulphate (SDS) with the monolayer of octadecylamine, octadecyl alcohol, and nylon 6 spread on the surface of the soln., has been measured by tracing sulphate sulphur tagged with ^{35}S in an apparatus previously described (*Ibid.*, **31** (1958) 532). The results suggest that the mechanism of the strong interaction of the long-chain amine monolayer with SDS is different from that of the monolayer of long-chain alcohol or nylon 6 with it (*Ibid.*, **32** (1959) 404, 645).   H.H.H.

Colloid Chemical Studies on Starching Materials. III— Dynamic Viscosity and Dynamic Rigidity of Starch Solutions
M. Nakagaki and K. Muragishi   *Bull. Chem. Soc. Japan*, **34** (Mar 1961) 316–319 (*in English*)

The frequency dependence of dynamic viscosity and of dynamic rigidity of 3% and 5% starch pastes were measured for potato (I), sweet potato (II), and rice (III) starch at 30°C. The results were compared with Maxwell's four-element model and Rouse's model, but neither fitted the experimental data. It was found that the rigidity of II was much greater than that of I, and both the rigidity and viscosity of III were smaller than those of I and II. A structural change of the paste depending on the frequency is suggested.   H.H.H.

PATENTS

Spinning Lubricant
Emery Industries   *USP* 2,944,920 (7 Nov 1955)

A solution of 5–50% by weight of polymerised fatty oils in oil is a good lubricant for carding, spinning, etc. and is more readily scoured out of the yarn or fabric than ordinary textile oils or soaps.   C.O.C.

Emulsifying Water-insoluble Organic Liquids
General Aniline   *USP* 2,946,721 (17 Dec 1954)

A mixture of (1) a water-soluble non-ionic surfactant (50–90 parts by wt.) containing a polyoxyalkylene chain of ≮ 2 alkenoxy groups and derived from an alkylphenolic compound (Alk of 4–24 C), and (2) the cyclohexyl-ammonium salt of an alkylbenzene sulphonic acid (50–10) containing ≮ one Alk of > 2 C, is soluble and stable in water-insoluble organic solvents, the solution when stirred into water readily producing a stable oil-in-water emulsion.   C.O.C.

Sequestering Agent
Co-operatieve Verkorp-en Productiervereniging van Aardappelmeel en Derivaten "Avebe"

BP 866,840 (Holland 16 Aug 1957)

A mixture of a water-soluble polyhydroxy compound of > 3 C and having > 1 vicinal OH group, e.g. gluconic acid,

and boric acid or a water-soluble borate has strong sequestering action, especially for Ca, at pH < 11. The sequestering action is little reduced and may even be improved at the boil.   C.O.C.

Sequestering Agent from Sucrose
Union Starch & Refining Co.

BP 868,802 (U.S.A. 2 Apr 1957)

Sucrose is converted into invert sugar by treatment with acid. The liquor is neutralised and treated with NaCN. The resultant cyanhydrins are hydrolysed, and the ammonia formed is removed to leave a mixture of Na glucon and fructoheptanoic acids. This mixture is an effective sequestering agent for the hardness-causing compounds in water and is especially suited for use in alkaline solutions at high temperatures.   C.O.C.

Bis(1 - alkyl - 1 - hydroxymethyl)phosphinic Acids— Sequestering Agents
American Cyanamid Co.

BP 867,883 (U.S.A. 27 June 1957)

Compounds of formula—

$$\left(\begin{matrix} OH \\ | \\ R \cdot C \\ | \\ COOY \end{matrix} \right)_2 \overset{O}{\underset{\|}{P}} \cdot OY$$
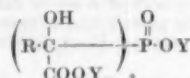
(R = Alk of 1–17 C; Y = H, alkali metal or alkaline-earth metal), e.g. trisodium bis(1-methyl-1-carboxy-1-hydroxymethyl)phosphinate, are sequestering agents particularly useful for the selective removal of heavy-metal ions in presence of alkali-metal and alkaline-earth-metal ions. Thus they permit the removal of ferric iron from the alum used in paper sizing.   C.O.C.

Rosin Products as Surfactants
Kodak   *BP* 867,842 (U.S.A. 9 Aug 1956)

Rosin and its derivatives, when treated with an iso-cyanatocarboxylic ester so as to form a –CO·NH– link, yield products having surface-active properties. These products, when added to coating compositions for paper, much improve the uniformity of the coating.   C.O.C.

Antistatic Agents
Vereinigte Glanzstoff-Fabriken

BP 867,820 (Germany 13 Dec 1958)

Compounds of formula

$$OP(OR^1)(HNR^2)_2, OP(OR^1)_2HNR^2, OP(OR^1)(NR^2_2)_2,$$
$$\text{and } OP(OR^1)_2NR^2R^3$$
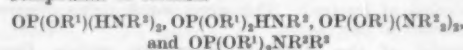
(R^1 and R^2 = Alk, Ar, aralkyl or hydroaryl), e.g. diethyl-stearylamidophosphate, are good antistatic agents for hydrophobic fibres, to which they are applied from solution in an organic solvent.   C.O.C.

Antistatic Agent for Polyamide Fibres
Vereinigte Glanzstoff-Fabriken

BP 869,086 (Germany 7 Jan 1959)

Treatment with an aqueous solution or dispersion of a compound of formula

$$\begin{matrix} OC_nH_{2n+1} \\ | \\ O:P-O(CH_2CH_2O)_xH \\ | \\ O \\ | \\ H_{2n+1}C_mNH_2 \end{matrix}$$
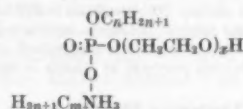
($x > 0$; $n = 3$–8; $m = 10$–20), are good antistatic agents for use in the processing of polyamide fibres, especially those prepared from caprolactam.   C.O.C.

Hardenable Condensates of Modified Ureas and Aldehydes as Softening, Water-repellent, and Wet-strength Agents
FH   *BP* 868,080 (1 Nov 1957)

A polyamine containing a reactive amino groups ($n > 2$) is treated with > 1 mol. of a substance which introduces urea groups into the amine so that ≮ 2 and ≯ $n - 1$ of these groups are introduced into the polyamine. The reaction product is then treated with a long-chain aliphatic compound containing a group which reacts with amine or carbonamide groups. Finally the resulting urea compound is treated with an aldehyde to convert it into a methylol or substituted methylol compound. The products render fibrous materials (e.g. textiles, paper, and leather) soft, water-repellent, resistant to abrasion, and of improved wet-strength.   C.O.C.

Polyfluoro-iodo Compounds— Water- and Grease-repellent Agents

Minnesota Mining & Manufacturing Co.

BP 865,858 (U.S.A. 18 Apr 1956)

A compound $Y(CF_2)_m CH_2I$ ($Y = H$ or F; $m =$ whole number) is treated in presence of an active source of free radicals with an ethylenically unsatd. aliphatic compound having at least one free H atom attached to C of the ethylenic link, the remaining valences of the ethylenic C atoms being satisfied by a combination of monovalent radicals, the algebraic sum of the Hammett *sigma* (para) parameter values of which is $>$ 0·2 (these values are described in *Chem. Reviews*, **53** (1953) 219–233). The product is oleo- and hydro-phobic.   C.O.C.

Fluorocarbon-substituted Triazine–Formaldehyde Condensates for Imparting Oil- and Water-repellency

Minnesota Mining & Manufacturing Corpn.

BP 867,279 (U.S.A. 11 June 1957)

Compounds of formula

$$
\begin{array}{c}
X \\
| \\
C \\
N\diagup \ \diagdown N \\
\| \qquad \| \\
R^1 \cdot Q \cdot W \cdot C \qquad CY \\
\diagdown N \diagup
\end{array}
$$

(X and Y = amino or hydrazino; R^1 = perfluorinated Alk of 4–12 C; Q = divalent sulphonyl, carbonyl or methylene; $W = -NH(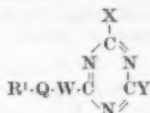CH_2)_n NH-$ or $-NR^2-$ ($n = 1$–12; $R^2 = H$ or Alk of 1–6 C) when $Q =$ sulphonyl, $-NH(CH_2)_m NH-$ or $-NH-$ ($m = 0$–12) when $Q =$ carbonyl, and $-(CH_2)_m O-$ or $-(CH_2)_m -NH-$ when $Q =$ methylene), e.g. 2,4-diamino-6-perfluorododecanoyl hydrazido-*s*-triazine, condensed with HCHO, yield products useful in obtaining good oil- and water-repellent finishes on textiles.   C.O.C.

Catalysts for Polymers containing Free $CONH_2$ Group

General Mills   *BP* 865,755 (U.S.A. 10 Dec 1958)

A mercuric salt of nitric or of an organic carboxylic acid is an excellent low-temperature catalyst for polymers containing free $CONH_2$ groups, particularly polymers of acrylamide and similar unsaturated amides. Thus cotton cloth was impregnated with a 5% aqueous solution of polyacrylamide, dried, treated in 5% aqueous mercuric acetate, rinsed, and air-dried. The treated pattern was much stiffer and resistant to folding than a control similarly treated but omitting the treatment with mercuric acetate.   C.O.C.

Chlorine-containing Organic Phosphorus–Nitrogen Compounds— Flame-resisting Agents

Joh. A. Benckiser G.m.b.H. Chemische Fabrik

BP 830,918 (Germany 28 Aug 1957)
BP 865,848 (Germany 4 June 1958)

The products obtained by treating a compound of formula $(R^1O)_3 POX$ with one of formula HNR^2R^3 ($R^1 =$ Alk or chloroalkyl; $X =$ Hal; $R^2 =$ an organic group; $R^3 = H$ or an organic group), e.g. $(ClCH_2CH_2O)_3 P(O)N \cdot (CH_2CH_2 \cdot CH_2)_2$, obtained from dialkylamine and di-2-chloroethylchlorophosphate, in equivalent amounts in presence of an aqueous inorganic base, are good flame-resisting agents.   C.O.C.

Fire-retardant Finish

Monsanto Chemical Co.   *USP* 2,749,233 (27 Jan 1953)
USP 2,949,385 (27 Jan 1953)

Phosphorus oxychloride is treated with ammonia at 155–200°C and the product treated with 10–35% of its weight of water at 80–150°C under $<$ 2 atm. pressure. The product gives a permanent flame-resistant finish on cellulose and its derivatives, e.g. cotton, cellulose acetate, cellulose nitrate, and rayon, without affecting their appearance or handle. The product may be used in conjunction with many synthetic resins.   C.O.C.

Flameproof Finish

Thermoid Co.   *USP* 2,948,641 (22 July 1957)

An aqueous composition containing a fluoborate, an inert texture-imparting agent, and a plasticiser is used. Thus a composition for flameproofing fabric of an asbestos-cotton blend (83/17) comprises water (100), Na fluoborate (3·0), Decerosol P (wetting agent) (0·3), finely ground mica (texture-imparting agent) (3·0), and an aqueous emulsion

(3·0) containing 85% tricresyl phosphate, and 5% diglycol stearate.   C.O.C.

Interaction between Acid Wool Dyes and Nonyl Phenol–Ethylene Oxide Derivatives (IV p. 452)

Effects of Softeners on Thermoplastic Resins (XIII p. 472)

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Quinones. I— Syntheses of Acetyl-*p*-benzoquinones and 2-Acetylnaphthoquinone

E. Kurosawa   *Bull. Chem. Soc. Japan*, **34** (Mar 1961) 300–304 (*in English*)

Acetyl-*p*-benzoquinone deriv. are expected to show interesting chemical and biological properties owing to the conjugation of the acetyl carbonyl group with the quinone double bond. The syntheses of 2-methyl- and 2,3-dimethyl-5-acetyl-*p*-benzoquinone and of 2-acetyl-1,4-naphthoquinone are now described via the oxidation of the corresponding acetylhydroquinone deriv. by Ag_2O and anhyd. K_2CO_3 in dry benzene. The infrared spectra are reported, and indicate the existence of a large M-effect which is confirmed by the relative low oxidation–reduction potential as obtained by the polarographic method.   H.H.H.

Tropylium Cations and their Effect on the Relation between Colour and Constitution of Aromatic Systems

E. Heilbronner   *Chimia*, **15** (Jan 1961) 35–39

General discussion of chemical and physical data of compounds containing the tropylium cation, e.g.

$$
\text{[ ]} \oplus
$$

and of the relation of colour to constitution in these compounds.   A.T.P.

Isolated Chromophoric Systems. XXXII— Colour Effects in Arylamides of *p*-Nitro-α-phenylcinnamic and α-(*p*-nitrophenyl)-cinnamic acids

A. V. Belotsvetov   *Zhur. obshch. khim.*, **31** (Jan 1961) 59–68

The effect of R^1 and R^2

$$
O_2N-\text{⟨ ⟩}-CH=C-CO-NH-\text{⟨ ⟩}-A \qquad (R^1)
$$

(I)

$$
\text{⟨ ⟩}-CH=C-CO-NH-\text{⟨ ⟩}-A \qquad (R^2, NO_2)
$$

(II)
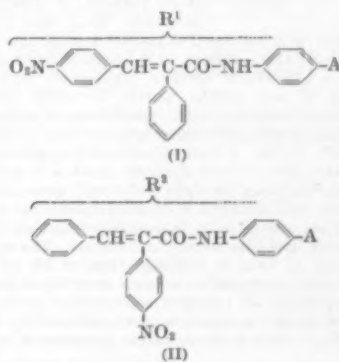
(A = OH, OCH_3, or $N(CH_3)_2$) on colour in I and II has been investigated spectrophotometrically in ethanol (220–600 mμ) and also by measuring absorption spectra (400–700 mμ) of I and II in powder form. A comparison of optical data with those of corresponding p-nitrocinnamoyl arylamides (III) shows that the introduction of an additional benzene ring in the α-position of III causes a hypsochromic shift of the absorption curves in the visible and ultraviolet regions and increased absorption in the short-wave region. These effects are more pronounced in the case of II. Analogous results have been obtained in absorption work on powdered I and II.   G.J.K.

Role of the Triplet State in Dye Photochemistry

G. Porter   *Chimia*, **15** (Jan 1961) 63

Of the two principal excited electron states in dye photochemistry, the lowest excited singlet, S_1, usually determines the colour of the dye, whilst the lowest triplet, T_1, has no effect on colour. The basic problems concerned

with photochemical reactions of dyes, e.g. fading, photo-tendering of fibres, are discussed.   A.T.P.

Interaction between Acid Wool Dyes and Nonyl Phenol-Ethylene Oxide Derivatives
B. R. Craven and A. Datyner

J.S.D.C., **77** (July 1961) 304–312

Relation between Constitution, Colour, and Light-fastness of Basic Dyes of the Indazole Series on Acrylic Fibres
R. Sureau   Chimia, **15** (Jan 1961) 195–203

The basicity and light fastness on Orlon of the dyes examined decrease in the order amino-6, -4, -7, and -5 indazole. On Courtelle and Crylor the relations were less exact.   E.S.

Relation between Colour and Constitution of Simple Nitro Dyes
E. Merian   Angew. Chem., **72** (1960) 766–770
Chem. Abs., **55** (15 May 1961) 9323

Preparation of a series of compounds 4,3-RNH(O₂N)· C₆H₃SO₂N(CH₂CH₂OH)₂ is described. When R = phenylazophenyl the compound has a high extinction coefficient (ϵ) in alcohol but it is of no practical interest as a dye because of its poor fastness to light. Substitution of 4-ether groups had practically no influence on ϵ in the visible spectrum but yielded disperse dyes of good fastness to light on cellulose acetate and polyester fibres. 2-Nitro-4-phenylazo-4′-(hydroxyethoxy)diphenylamine dyed polyester fibres yellow of good fastness to light, but its light fastness on cellulose acetate was poor because optically it resembles a *p*-aminoazo rather than a nitro dye. Replacing the NH₂ in 2,4-dinitroaniline by an arylamino group caused ϵ to increase by about 20%. Comparison of the substituted 2,4-dinitroaniline with the first series of compounds showed that the less electron-accepting SO₂NH₂ group shifted the absorption maximum by 60 mμ compared with the NO₂ group, but at the same time reduced ϵ_{max}. The colour intensity of both types of dyes was therefore similar. Dyes of the first series are better technically because of their fastness to water and because the substituted 2,4-dinitro anilines yield dyeings whose colour changes under the action of light.   C.O.C.

Acid Dyes derived from 1-Amino-2-naphthol-4-sulphonic Acid
A. I. Levchenko   Izvest. Vysshikh. Ucheb. Zavedeniĭ,
Khim. i Khim. Tekhnol., **3** (1960) 920–923
Chem. Abs., **55** (1 May 1961) 8865

Oxidation of 1-amino-2-naphthol-4-sulphonic acid (I) first yielded the yellow 1,2-iminonaphthoquinone-4-sulphonic acid, which further condensed with I with elimination of one sulpho group to yield an orange quinone-imino acid dye (II). Oxidation of a mixture of 1 and 2-amino-8-naphthol-6-sulphonic acid (III) yielded the brown di-Na 1-hydroxydibenzo[a,j]phenazine-3,9-disulphonate (IV). These dyes on wool and silk showed satisfactory fastness to light and wet processing. The colour value and fastness of II were somewhat inferior to those of other orange acid dyes and exhaustion was poor. IV was faster and was readily exhausted. The absorption maximum of II was at 470 mμ, that of IV at 523 mμ. They were also prepared *in situ* on wool by impregnating with a dispersion of I mixed with other aminohydroxy sulphonic acids and aromatic amines, followed by oxidation with NaClO₃ in acid medium and in presence of a catalyst.   C.O.C.

Influence of Spatial Factors on Properties of Dyes Containing the Diphenyl Nucleus—XV
B. M. Krasovitskiĭ, L. M. Litvinenko, N. J. Titarenko, and N. F. Levchenko
Ukrain. khim. zhur., **27**, No. 1 (1961) 94–97

Monoazo dyes obtained from 4-aminodiphenyl were compared with similar dyes in which two benzene rings were linked by a number of different groups, e.g. derivatives of diphenylmethane, dibenzyl, *trans*-stilbene, diphenylacetylene, azobenzene, and *p*-terphenyl. The absorption spectrum in aqueous solution is determined by the nature of the linking group.   L.S.L.

Studies on Chrome Complex Dyes
R. Asaoka, H. Suzuki, and H. Iida
Rept. Govt. Chem. Ind. Research Inst. Tokyo,
56 (Mar 1961) 22–27

The chromium complexes of four monoazo dyes obtained from diazotised 2-aminophenol-4-sulphonamide and 2-aminophenol-4-methylsulphone by coupling each with β-naphthol and 1-phenyl-3-methyl-5-pyrazolone have been studied. Each dye was converted into the corresponding 2:1 chromium complex by the action of aqueous ammonium chromosalicylate. The crude complexes so obtained were dissolved in ethanol and passed through an alumina column when it was found that the 2:1 complex passed into the filtrate. In each case, however, the corresponding 1:1 complex, obtained as a by-product, was absorbed by the alumina. Experiments on the paper chromatography of the 2:1 and 1:1 complexes (the latter prepared by an alternative route) are described, and R_f values in a 2:1 mixture of butyric acid and water are given. Wool has been dyed with purified samples of each 1:1 and 2:1 complex, and the colours of the dyed wool samples have been determined spectrophotometrically.   I.G.L.

Relation between Structure of Azo Dyes and Rate of Diffusion in Cuprammonium Rayon Yarn
B. N. Mel'nikov, B. M. Krasovitskiĭ, P. V. Morÿganov, and T. D. Zakharova
Tekhnol. tekstil. prom., No. 6 (19) (1960) 120–124

The speed of diffusion of certain disazo dyes (I–III)
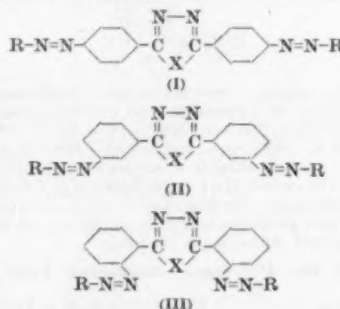
where (X = O or S) which differ in relative positions of azo groups and in character of hetero-atom, have been investigated. The rate of diffusion was measured at 50 and 70°C. The breaking of conjugation between azo groups increases the diffusion coefficient. Transfer of the azo group from *para*- to *ortho*-position in relation to the heterocyclic ring will also increase the rate of diffusion. This is explained by steric hindrance.   L.S.L.

Relation between Structure and Light Fastness of Insoluble Azo Dyes
P. P. Karpukhin and Yu. V. Naboĭkin
Ukrain. khim. zhur., **26**, No. 6 (1960) 736–739

The energy involved in photochemical reactions occurring during exposure of 18 insoluble azo dyes to light was calculated. The light fastness of such dyes depends on the electron-donating properties of nitrogen in the azo group. NO₂ or OCH₃ *ortho* to the azo group will increase the light fastness. Dyes having substituents *para* to the amido group (in naphthol residue) will show greater light resistance than similar dyes having substituents in *ortho*- and *meta*-positions.   L.S.L.

Syntheses of Unsymmetrical Azo Compounds
F. Suckfüll and H. Dittner
Chimia, **15** (Jan 1961) 137–147

syn-Diazosulphonates R¹·N:N·SO₂⊖ combine with diazonium compounds R²·N⊕:N to give R¹·N:N·SO₂·O·N:N·R², which loses N₂ and readily gives the unsymmetrical azo compound R¹·N:N·R². R¹ may be a polynuclear as well as a simple benzene-series radical; R² is a naphthalene radical, or a benzene radical free from electronegative substituents, e.g. NO₂. Thus the *syn*-diazosulphonate from sulphanilic acid, with diazotised 1-naphthylamine, gives
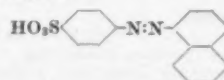
Conditions were also established under which *syn*-diazotates combined with diazonium salts with introduction of an o-hydroxy group. Thus the *syn*-diazotate

from p-benzoylaminoaniline combined with diazotised 2-naphthylamine-3,7-disulphonic acid to give
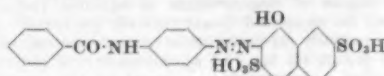
Many azo compounds difficult to prepare by conventional methods can be made by these methods.   E.S.

Preparation of Azo Pigments by Continuous Processes
H. Nakaten   *Chimia*, 15 (Jan 1961) 156–163
Apparatus for continuous azo coupling in the laboratory and on the technical scale is described. Rate of coupling is an important factor in determining crystal form and size, so that continuous coupling may confer different pigment properties on the product than those obtained in batch-wise manufacture. A coupling vessel of 40 m³ gives an output of ca. 20 tons of Hansa Yellow G (C.I. 11680) per month operating batch-wise; a coupling vessel of 400 l. operating continuously is capable of a similar output.
        E.S.

Food Dyes
S. Fujii and M. Kamikura
I— Low-sulphonated Dyes in Ponceau 3R (C.I. Food Red 6)
Eisei Shikenjo Hōkoku, No. 77 (1959) 167–174
In six commercial samples of the dye, 0·1–7·0% of Na 1-(2,4,5-trimethylphenylazo)-2-naphthol-6-sulphonate was found as an impurity.
II— Subsidiary Dyes in C.I. Acid Red 106
Ibid., 447–480
Chem. Abs., 55 (29 May 1961) 10731
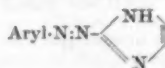Paper chromatography revealed the presence of at least three contaminating unidentified dyes.   C.O.C.
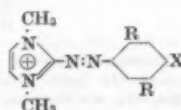
New Syntheses for Diazostyryl Dyes
H. Baumann and J. Dehnert
        Chimia, 15 (Jan 1961) 163–168
Using imidazole as coupling component, mono- and dis-azo dyes of the type

were prepared. Basic dyes of the type

(R = N or OCH₃; X = subst. amino residue, e.g. NH·C₆H₅) were prepared by quaternising compounds where X = Cl, followed by condensation with suitable amines.   E.S.

Fluoro-substituted Dyes
H. Hopff   *Chimia*, 15 (Jan 1961) 193–194
The influence of the introduction of a fluorine atom into azo and azoic dyes and pigments, and into vat dyes and phthalocyanine pigments is reviewed. It sometimes gives increased light fastness.   E.S.

Mechanisms of Diazotisation at Intermediate Acidity
B. C. Challis and J. H. Ridd
        Proc. Chem. Soc., (May 1961) 173–174
Following the mechanisms already discussed for media of very low (cf. *J.C.S.*, (1958) 58) and high acidity (cf. *Proc. Chem. Soc.*, (1960) 245), the intermediate region of perchloric acid concn. 0·5–3·0 M is now considered. The complex dependence of reaction rate on acidity is found to arise mainly from a medium effect of perchloric acid similar to that of sodium perchlorate, combined with one new kinetic term due to a reaction involving nitrosation of the protonated amine in such a way that the proton being displaced is still present in the transition state, and equivalent to an S_E2 reaction at a protonated nitrogen atom.   H.H.H.

Reactions of Diazonium Salts with Phenols and Amines in Non-aqueous Media
W. Bradley and J. D. Thompson
        Chimia, 15 (Jan 1961) 147–155
Benzene-soluble diazonium salts are prepared by applying the usual diazo reaction to alkoxyanilines having n-heptyl, n-decyl, and cetyl groups in the alkoxy substituents. The coupling reaction in benzene is not essentially different from that in aqueous solution, but by-products may arise from radical reactions if the conditions of coupling favour the formation of diazo ethers or diazoamino compounds at the first stage of the reaction.   A.T.P.

Reactive Methylene Compounds. VII— Condensations with Benzenediazonium Salts
H. G. Garg   *J. Indian Chem. Soc.*, 38 (Apr 1961) 211–212
The dyes obtained by coupling benzenediazonium chloride and its 4-methoxy-, 2-, 3-, and 4-chloro-, 4-bromo-, 3- and 4-nitro-, 4-methyl-, 2-chloro-4-nitro- and 2-nitro-4-chloro-deriv. respectively with 3,4-dichloro-(I) and 2-chloro-5-(II) methylbenzoylacetone, and with ω-anisoyl and ω-benzoyl-3,4-dichloroacetophenone (III), are described. The syntheses of I, II, and III are reported.
        H.H.H.

Photodecomposition of Diazonium Salt Solutions
W. E. Lee, J. G. Calvert, and E. W. Malmberg
    J. Amer. Chem. Soc., 83 (20 Apr 1961) 1928–1934
Photochemical decompositions of p- and m-nitrobenzene-diazonium salt soln., stabilised as their stannic chloride, zinc chloride, or fluoroborate complexes, in various solvents and in presence of free-radical traps have been studied. The reaction mixtures were separated by use of silicic acid chromatographic columns. Results showed that the dominant primary process in alcoholic soln. is nitrophenyl radical formation. Nitrobenzene, α- and β-arylethanol and butanediol are formed, but no dinitrodiphenyl could be detected. In aq. soln. carbonium ion appeared to be exclusively formed on absorption of light. Results of photolysis in rigid media at −196°C suggested the intermediate formation of a long-lived triplet state at low temperatures. The mechanisms of decomposition in alcoholic and aq. soln. are discussed and preference is given in the latter case to the presence of a photo-excited state rather than free radicals as postulated by Boudreaux and Boulet, *ibid.*, 80, 1588 (1958). Finally, the preparation of aromatic iodo- and bromo- deriv. via the photochemical decomposition of diazonium salts is presented and claimed to give improved yields. A possible solution to the problem of synthesis of aromatic hydroperoxides is also reported.   F.J.

Transformations of Triphenylmethane Dyes in Acid Solutions. II— Complex Acid–Base Equilibria
O. F. Ginzburg and P. M. Zavlin
        Zhur. obshch. khim., 31 (Jan 1961) 75–80
Acid–base equilibria in aq. soln. of Malachite Green (C.I. Basic Green 4) containing substituents in *para*- and *meta*- positions (m-OCH₃, m-Cl, or p-Cl) have been investigated spectrophotometrically (430–620 mμ). It is found that for the pH range 0–4 the concn. of doubly charged conjugated carbonium ions is small and it is therefore assumed that in such soln. there are practically only colourless ammonium ions and singly charged coloured conjugated carbonium ions.   G.J.K.

6-Aminoselena-2′-cyanines
K. Meyer and K. H. Bauer
        Z. wiss. Phot., 54 (1960) 171–173
        Chem. Abs., 55 (29 May 1961) 10157
The absorption maximum of 3,1′-diethyl-6′-acetamido-selena-2′-cyanine iodide is at 514 mμ, 14 mμ bathochromic to that of the dye unsubstituted in the 6′-position of the quinoline nucleus, and that of 3,1′-diethyl-6′-amino-selena-2′-cyanine iodide is at 522 mμ.   C.O.C.

Cyanine Dyes. Synthesis of 1-(4-Ethoxyphenyl)-7,8-benzoquinaldinium Perchlorate and its Derivatives
G. T. Pilyugin and N. A. Tsvetkova
Uchenye Zapiski Cheinovitsk. Gosudarst. Univ., Ser. Khim., 21 (4) (1956) 59–67
        Chem. Abs., 55 (1 May 1961) 8412
Condensation of secondary aromatic amines with paraldehyde under suitable conditions led to cyclisation

and formation of quinaldine derivatives having aryl radicals on the heterocyclic N. Study of the arylated quaternary salts showed that presence of aryl radicals enhanced the reactivity of the salts compared with that of the alkyl analogues. The effect of aryl groups manifested itself also in bathochromic shifts of the absorption maxima of the dyes.   C.O.C.

Merocyanines and Cyanines derived from 2-Hydroxy- and 2-Mercapto-thiazoles. Their Synthesis and Absorption Spectra
E. B. Knott   Chimia, 15 (Jan 1961) 106–109

General syntheses of the title compounds are discussed. Thus, condensation of 2-(2'-acetanilidovinyl)-3-ethyl-benzoxazolium iodide (I) and 2-hydroxy-4-methyl-thiazole in presence of alcoholic sodium ethoxide and pyridine gave a yellow solution (normal dye), rapidly degraded on further heat giving an intense blue–green solution of trinuclear dye (due to nucleophilic activity of the 4-methyl group condensing with a second mol. of I). Absorption of the various types of dyes described is discussed, e.g. cyanines absorb at much shorter wavelengths than the parent merocyanines, owing to an increase in the energetic asymmetry of the hybrid.   A.T.P.

New Flavylocyanines and Merocyanines
T. Photakis   Chim. & Ind. (Paris), 84 (1960) 908–911
Chem. Abs., 55 (15 May 1961) 9394

A review of the synthesis of asymmetric flavylocyanines and merocyanines from flavylium perchlorate with heterocyclic end-groups in acetic acid, using anhydrous Na acetate as accelerator.   C.O.C.

Synthesis of Merocyanine Dyes, Derivatives of Rhodanine. I— Exchange of the Aniline Group in the Pentamethinemerocyanines for a Secondary Amine Group
J. Oszczapowicz and J. Świderski
Roczniki Chem., 34 (1960) 959–964
Chem. Abs., 55 (29 May 1961) 10418

Merocyanine dyes are obtained by exchanging the aniline group at the end of the polymethine chain in pentamethinemerocyanine dyes derived from rhodanine, for a secondary amino group. A secondary amine is added to 1 mole glutaconic dialdehyde dianil hydrochloride and 1 mole 3-alkylrhodanine in boiling absolute alcohol, the mixture boiled for several hours, stored for 1 day, and the crude dye filtered off and recrystallised from dry CHCl$_3$ or C$_6$H$_6$.   C.O.C.
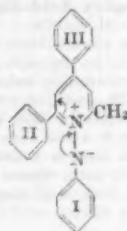
Influence of Solvents on Organic Dyes
K. Dimroth   Chimia, 15 (Jan 1961) 80–86

General discussion of various effects of solvents (e.g. on λ_{max}) with special reference to compounds

where rings I to III may be substituted.

Discussion on the above paper
L. G. S. Brooker   Ibid., 87–89

The "solvent sensitivity curve" evidence of Dimroth for the existence of an "isoenergetic point" of merocyanine dyes is discussed. It is indicated that certain merocyanines give two primary absorption bands.   A.T.P.

Infrared Absorption Spectra of Polymethine Dyes
M. Pestemer   Chimia, 15 (Jan 1961) 31–35

In the one-dimensional π-electron clouds of double-bonded conjugated chains, especially those with charge resonance (as in polymethine dyes), only the bonds between the extreme atoms have the same double bond character. Strong infrared-active valency oscillations are present in the vicinity of the double bonds (between 1480 and 1590 cm^{-1}).   A.T.P.

Mesomeric Degradation of Polymethine Dyes
A. van Dormael   Chimia, 15 (Jan 1961) 67–74

The degree of degeneration of cationic polymethine dyes can be expressed quantitatively by certain optical data, principally (a) variation of optical increments due to a vinyl group, (b) λ_{max} of an asymmetrical polymethine dye perfectly degenerated is equal to the arithmetical mean of λ_{max} of the corresponding two asymmetric colours, (c) λ_{max} of an ionised colour is related to variations in polarity of solvents. The above are discussed with relation to known polymethine dye structures and also to a new type, the phosphinines (characterised by an electron-donating group $\rangle$C=P$\langle$ at one end of the chain, and an electron-accepting group $\rangle$C-P$\langle$ + at the other), and related dyes described as the phosphocyanines and merophosphinines.   A.T.P.
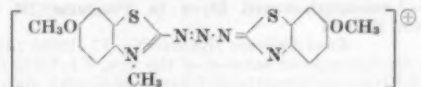
Azamethines— Basic Dyes for Polyacrylonitrile Fibres
J. Voltz   Chimia, 15 (Jan 1961) 168–176

The relation between structure and colour of many symmetrical and unsymmetrical mono-, di-, and tri-azacyanines and of tricarbocyanines is discussed. Light-fastness of dyeings on polyacrylonitrile fibres of yellow and red symmetrical triazacyanines is very high, e.g.

which is a red, the OCH$_3$ groups having a marked batho-chromic effect.   E.S.

Influence of Bridging on Colour of Polymethine and Azamethine Dyes
G. E. Ficken and J. D. Kendall
Chimia, 15 (Jan 1961) 110–115

Introduction of a substituent into the polymethine chain of cyanine dyes markedly affects its photographic properties. Recent work on many chain-substituted dyes is described, with reference to (a) preparation, e.g. the pentamethincyanines bridged by a saturated chain in the $\beta\beta'$-position
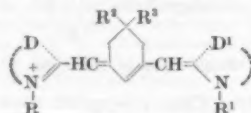
from cyclohexane-1,3-diones and their reaction products with mercaptans, and (b) light absorption, which is affected in two ways, viz. an electron effect due to the bridge and sometimes a steric effect.   A.T.P.

Structure and Chemical Properties of "Methylene Base" derived from 2-Methylbenzothiazole
H. Larive and R. Dennilauler
Chimia, 15 (Jan 1961) 115–126

Quaternary salts of 2-alkylbenzothiazole may yield acetylated aminothiophenols by hydrolytic ring opening, or a 2-alkylidene base by loss of the ion and a proton attached to the α-C adjacent to N. The accepted "cyclo-butane dimer" molecular structure of the methylene base was shown to be incorrect and an asymmetrical structure
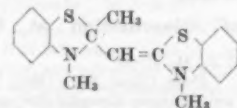
was shown to account for the observed chemical properties and to explain the mechanism of dye formation, especially of meso-substituted merocyanines and tri-methylcyanines, hitherto unexplained.   A.T.P.

Thiazolocarbocyanines containing an Aryl Group in the Thiazole Nucleus. VIII— Asymmetrical Thiazolocarbocyanines
E. D. Sych   Ukrain. khim. zhur., 27, No. 1 (1961) 83–87

Anilide- and acetanilide-vinyl derivatives of quaternary salts of heterocyclic nitrogenous bases were prepared. These

were treated in pyridine in presence of a small amount of acetic anhydride to give asymmetrical carbocyanines containing benzothiazole residues and also thiazole residues with polar substituents. Reaction products were separated as iodides and recrystallised from alcohol. Absorption spectra of new dyes with the general formula

$$\underset{\substack{A}}{R^2\text{---}} \overset{S}{\underset{\underset{C_2H_5}{N}}{}} \text{+C---CH=CH---CH=C} \quad X^- \quad \underset{\substack{B}}{\overset{S}{\underset{\underset{C_2H_5}{N}}{}}} Ar$$

were compared with those of similar dyes having both residues AA and BB. The position of the absorption peak of the new dye is always lower than the arithmetical mean of AA and BB positions. This difference is called the "hypsochromic displacement" and is generally < 15 mμ.
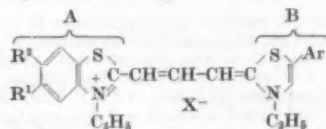L.S.L.

Chelate Stabilities of Certain Oxine-type Compounds. II— 4-Hydroxybenzothiazoles
T. J. Lane and A. Sam
J. Amer. Chem. Soc., 83 (20 May 1961) 2223–2225
Acid dissociation constants of 4-hydroxybenzothiazole, 2 - amino - 4 - hydroxybenzothiazole, 2 - methylamino - 4 - hydroxybenzothiazole, and 2-amino-4-hydroxy-7-methyl-benzothiazole were determined in 50% (v/v) p-dioxan at 25°C, and the chelate stability constants of these ligands with divalent Cu, Pb, Ni, Co, Zn, and Cd cations were obtained by the Calvin–Bjerrum potentiometric titration technique. Stability constants of 4-hydroxybenzothiazole chelates are lower than those of the corresponding chelates of 8-hydroxyquinoline, and this is explained by a larger nitrogen–oxygen distance and unfavourable electron orientation on the donor nitrogen atom of the former cpd. Stability values are higher than those of 4-hydroxy-benzimidazoles and 4-hydroxybenzoxazoles. The increase is attributed to the influence of the larger sulphur atom in the 1-position causing the 3-nitrogen atom to be closer to the chelating oxygen thereby reducing strain in the chelate ring structure.
F.J.

Sulphuric Esters of Leuco Vat Dyes
H. Schenkel   *Chimia*, 15 (Jan 1961) 203–207
A study of the rates and mechanisms of the reactions of reduction and esterification of indigoid and anthraquinone vat dyes.
E.S.

Constitutional Problems Concerning Anthra-quinonoid Vat Dyes
K. Venkataraman
J. Indian Chem. Soc., 38 (Apr 1961) 197–210
Subjects reviewed in this Presidential Address to the 37th Annual General Meeting of the Indian Chem. Soc. are: chromatographic analysis, absorption spectra of leuco derivatives, steric hindrance in dibenzanthronyls and dibenzanthrones (in particular in Caledon Jade Green, C.I. 59825), nitration of Dibenzanthrone (C.I. 59850), constitution of Bally's "Benzanthronequinoline" and of Cyananthrene (C.I. 68705), anthrimides and carbazoles, Cibanone Yellow R (C.I. 69705), and Orange R (C.I. 69700) (the marked photochemical activities of which have made possible their use in investigations on the accelerated oxidation of cellulose by light), and reduction of quinones to hydrocarbons. 55 References.   H.H.H.

Synthesis of Indanthrone from Leuco-2-Amino-anthraquinone
Y. Bansho, S. Suzuki, and I. Saito
Rept. Govt. Chem. Ind. Research Inst., Tokyo
56 (Apr 1961) 165–172
Experimental data are assembled concerning the various stages of the title synthesis. The paper, which is entirely in Japanese (with summary in German), is prefaced by a brief historical account of the discovery of Indanthrone (C.I. 69800). 19 References.   H.H.H.

Substituted Analogues of Phthalocyanine (C.I. 74100). Synthesis and Properties
V. F. Borodkin and R. P. Smirnov
Izvest. Vysshikh Ucheb. Zavedeniĭ, Khim. i Khim Tekhnol., 3 (1960) 915–919
Chem. Abs., 55 (15 May 1961) 9371
Describes the preparation of 11 derivatives of phthalo-cyanine. Introduction of amino groups into the isoindole radical of analogues of phthalocyanine containing one benzene radical resulted in a shift of the absorption maximum towards longer wavelengths, the displacement becoming more marked with increase in the number of amino groups. Introduction of nitro groups also resulted at first in displacement of the absorption maximum towards longer wavelengths, and one NO_2 group caused greater shift than one NH_2 group. Accumulation of NO_2 groups resulted in a remarkable shift of the absorption maximum towards the short-wave region. This was apparently caused by lowering of the electron density in the ring. In analogues having 2 benzene radicals, presence of two amino or nitro groups in the isoindole radical did not affect the absorption spectrum (λ_{max} 350 mμ). Presence of 8 chlorine atoms shifted the maximum towards longer wavelengths (λ_{max} 480 mμ instead of 350 mμ). The absorption maxima of analogues of phthalocyanine or naphthalocyanine containing C_6H_6 and C_5H_5N radicals were at longer wavelengths than those of analogues having two benzene radicals (λ_{max} 385 and 396 mμ, instead of 350 mμ). This is probably caused by participation in the conjugation of the undivided pair of electrons of the N atom of the C_5H_5N residue. Absorption spectra were measured in α-chloronaphthalene.   C.O.C.

Electron Spin Resonance of Copper Phthalocyanine
E. M. Roberts and W. S. Koski
J. Amer. Chem. Soc., 83 (20 Apr 1961) 1865–1867
The electron spin resonance spectrum of Cu phthalo-cyanine (C.I. Pigment Blue 15) in sulphuric acid at -137°C was studied. The nitrogen hyperfine structure was observed and nitrogen splitting was smaller in the region near $H_{||}$ than in the $H_{\perp}$ region. An explanation for this is proposed and a bonding parameter reflecting the nature of the copper–nitrogen bond is evaluated. Values of $g_{||}$ and $g_{\perp}$ were determined as 2·180 and 2·037, respectively.   F.J.

Synthetic Carotenoids for the Coloration of Food-stuffs
O. Isler, R. Rüegg, and P. Schudel
Chimia, 15 (Jan 1961) 208–226
Lycopene (pigment of tomatoes), torularhodin (red yeast), and canthaxanthin (edible mushroom) are being tested for suitability as colorants for foodstuffs. Synthetic β-carotene has been in commercial use since 1954, and β-apo-8′-carotenal (C_{30}) will shortly reach this stage. Syntheses based on citral are outlined.   E.S.

Alkanna tinctoria—a Source of Alkannin (C.I. Natural Red 20)
M. Tyllová, G. Dušinský, and A. Szokolay
Farmácia, 29 (1960) 274–277
Chem. Abs., 55 (15 May 1961) 9791
A. tinctoria, which grows in Slovakia, is a source of alkannin (C.I. 75530). Alkannin can be identified and estimated spectrophotometrically at 490 mμ, by polaro-graphy in solution buffered at pH 7, and by paper chromatography in methanol–acetic acid–water (80:5:15).   C.O.C.

Sources of Pigments in Ancient China
Y. Yonezawa   *Mem. Inst. Oriental Culture, Tokyo Univ.*, No. 11 (1956) 375–395
I.I.C. Abstr., 3 (1960) Abstr. No. 2558
Chem. Abs., 55 (15 May 1961) 8971
A list compiled from ancient books and documents is given of the sources of pigments in ancient China from pre-Ch'in to Tang periods. The sources are all in South China up to and during the Han period; after that places in North China, tropical countries, and Central Asia are mentioned. The pigments listed are Cinnabar (C.I. 77766), Malachite (C.I. 77420), Azurite (C.I. 77420), Red Lead (C.I. 77578), White Lead (C.I. 77597), Realgar (C.I. Pigment Yellow 39), and Orpiment (C.I. 77086). Lac Dye (C.I. Natural Red 25) and glue are also dealt with.   C.O.C.

Origin of Colour in Copper Red Glass
A. Ram, S. N. Prasad, and V. K. Vaish
Central Glass & Ceram. Research Inst., Bull (India), 7 (1960) 49–53
Chem. Abs., 55 (29 May 1961) 10831
Before heat-treatment the Cu is present as Cu^+ and not as Cu atoms.   C.O.C.
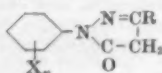
Colour Couplers

Kodak   *BP* 868,937 (U.S.A. 6 Sept 1956)

Colour couplers which are stable to heat and light in photographic emulsions and which yield dyes whose secondary light absorptions are low are of formula

(X = Hal; $n > 1$; R = Alk of 11–17 C) e.g. 1-(2,4,6-trichlorophenyl)-3-pentadecyl-5-pyrazolone.   C.O.C.

Water-soluble Non-diffusing Dye Components for Colour Photography

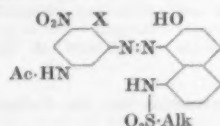Gasparcolor and G. B. Linden

USP 2,943,934 (8 May 1951)

Compounds of formula HNR^1R^2, H_2NR^2 and HOR^1 (R^1 = functional group rendering the compound capable of forming dyes; R^2 = H, Alk or Ar; R^3 = aromatic radical having an available coupling function), e.g. benzoylacet-(p-amino)anilide, 1-naphthylamine and 1,5-dihydroxynaphthalene, are rendered non-diffusing by treating them with compounds of formula $(-A(SO_2Cl)-)_n$ (A = organic radical; $n > 2$) e.g. chlorosulphonated polystyrene, to yield compounds of formula $(-A(SO_2Y)-)_n$, where at least some of the Y's = NR^1R^2, OR^1 or NHR^2, and some = Cl and after hydrolysis = an –O– cation group.   C.O.C.

Blue Metallisable Monoazo Dyes for Wool, etc.

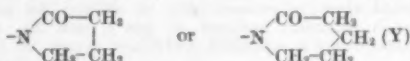FBy   *BP* 869,842 (Germany 5 Apr 1958)

Monoazo compounds

(Ac = acyl radical of carboxylic acid; X = OH or group converted to OH during metallisation; Alk is of 1–4 C) similar to those of *BP* 755,853 (J.S.D.C., **72** (1956) 545) are converted to Co or Cr complexes in substance or on the fibre to give blues. Thus 4-acetylamino-2-amino-4-nitrophenol is diazotised and coupled with 1-methyl-sulphonylamino-7-naphthol to give a blue monoazo metachrome dye which may be converted into the reddish blue 1 : 2 cobalt-complex.   E.S.

Metal(Chromium and Cobalt)-complex Monoazo Heterocyclic Dyes for Wool, etc.

S   *BP* 869,031 (Switzerland 20 July 1956)

The title dyes are neutral-dyeing 1 : 2 metal complexes of monoazo compounds, similar to those of *BP* 835,470 (J.S.D.C., **76** (1960) 512) and contain 1 or 2 substituents

Thus diazotised 2-amino-4-nitrophenol is coupled in presence of NaOH and Na_2CO_3 with 1-Y-7-naphthol, and the monoazo compound so formed is heated in formamide with chrome alum to give the Cr-complex which dyes wool, nylon, and leather black.   E.S.

Monoazo Disperse Dyes for Polyester Fibres

BASF   *BP* 870,677 (Germany 11 May 1957)

The title dyes are prepared by coupling diazotised p-nitroaniline and suitable derivatives thereof with tertiary amino derivatives of N-β-cyanoethylaniline, e.g. N-methyl-N-β-cyanoethyl-m-toluidine, the dyes being free from COOH or SO_3H groups, but optionally containing substituents, e.g. Alk, Hal, OAlk, $SO_2\cdot NH_2$, $CO\cdot NH_2$, etc. Thus diazotised p-nitroaniline coupled with N-butyl-N-β-cyanoethylaniline gives an orange.   E.S.

Azoic Dyes Containing Silicon

Union Carbide Corpn.   *BP* 870,467 (U.S.A. 29 Apr 1957)
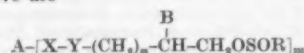
The dyes of *USP* 2,927,829 (J.S.D.C., **77** (June 1961) 260) are applied to cotton by the azoic method. Thus cotton impregnated with β-naphthol becomes bright red when immersed in a solution of diazotised β-(aminophenyl)-ethyltriethoxysilane.   E.S.
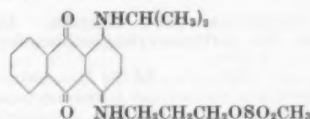
Anthraquinone Disperse Dyes

ICI   *BP* 870,948 (29 Oct 1958)

The above are

$$A-[X-Y-(CH_2)_n-\overset{B}{CH}-CH_2OSOR]_m$$

(A = subst. or unsubst. anthraquinone; B = H or OH; X = direct link, alkylamino or arylamino; Y = bridging atom or group; R = Alk, phenyl, tolyl or chlorophenyl; $m = 1–2$; $n = 0–2$, $n \neq 0$ when B = OH, and A, X, Y, and R are free from SO_3H and COOH). They are prepared by any of the known methods and dye synthetic-polymer fibres, especially polyamides and modified polyacrylonitrile, bright colours of excellent fastness to light and washing. Thus, 1-isopropylamino-4-γ-hydroxypropylaminoanthraquinone (5·1) and methane sulphonyl chloride (2·25) are reacted at 0–5°C for 3 h in pyridine (75). Water (750) is added and the blue dye

filtered. It has excellent fastness to light and washing on nylon. Similarly a bright greenish blue dye is obtained from p-toluenesulphonyl chloride and 1-methylamino-4-p-(βγ-dihydroxypropoxy)anilinoanthraquinone.   A.T.P.

Quinophthalones— Yellow Disperse Dyes for Polyethylene Terephthalate

FH   *BP* 870,680 (Germany 25 Sept 1957)

The condensates of aromatic *ortho-* or *peri-*dicarboxylic anhydrides with subst. or unsubst. 2-methylpyridines or 2-methylquinolines are greenish yellow to reddish yellow dyes for polyesters (or, after sulphonation, acid dyes) and have good fastness. Thus, 4-fluorophthalic anhydride is condensed with quinaldine to give a clear greenish yellow disperse dye.   A.T.P.

2,2′-Difluoroacedianthrone— Brown Vat Dye

Ciba   *BP* 870,143 (Switzerland 4 Oct 1956)

This dye is obtained by cyclising bis-(2′-fluoro-9′-anthronylidene-10′)-ethane or -ethylene. It is faster to light than the 2,2′-dichloroanalogue.   A.T.P.

Methine Dyes

FBy   *BP* 869,794 (Germany 23 May 1957)

Methine dyes for mordanted cotton, leather, cellulose acetate, polyamides, polyurethane, and polyacrylonitrile fibres are

(R^1, R^4, R^5, and R^6 = Alk; R^2 = H or Alk; R^3 = H, Alk or Ar; X = anion, any of benzene nuclei may be substituted). They are obtained by condensing 7-alkylindole-3-aldehydes in acid solution with 1,3,3-trialkyl-2-methylene indolines, or by condensing 7-alkylindoles with 1,3,3-trialkylindoline-2-methylene-ω-aldehydes or by reacting 7-alkylindoles and 1,3,3-trialkyl-2-methyleneindolines with a dialkyl, diaryl, or alkylaryl formamide and halides of P or H_3PO_4. Thus, the condensate of 1,3,3-trimethyl-2-methyleneindoline-ω-aldehyde and 2-methyl-7-ethylindole dyes polyacrylonitrile brilliant orange.   A.T.P.

Red to Brownish Red Thiazine Dyes

Gevaert Photo-Producten   *BP* 869,138 (11 July 1958)

Condensing a phenothiazine aldehyde with a compound containing a reactive methylene or methyl group yields thiazine dyes specially useful for photographic filters or antihalation layers and which are permanently bleached when the exposed material is developed and fixed. Thus 3 - formyl - 10 - methylphenothiazine treated with 1-(p-sulphophenyl)-3-heptadecyl-5-pyrazolone yields the magenta

$$H_3C(CH_2)_{16}-C\!=\!C\text{-}CH\text{-}$$

(structure with N, CO, S, N-CH₃, and SO₃H)

Photosensitising Dyes

Ilford   BP 867,592 (19 June 1958)

  Dyes of formula

(structure: Z— ring with CH₃, C-C(CHCH)ₙ:N, O-CO, D)

($n = 0$ or 1; D = atoms to complete a 5- or 6-membered ring; Z = one or more Alk, aralkyl, Ar, OH, alkoxy, NH₂, or substituted amino groups or H or Hal), e.g. the yellow 3-(benzothiazolyl-2)-7-hydroxycoumarin, have photosensitising properties.   C.O.C.

Merocyanine Dyes and Undissociated Cyanine Dyes

Kodak   BP 869,521 (U.S.A. 30 Aug 1956)

  The methyl group of dyes of formula

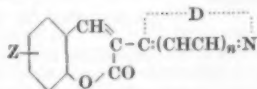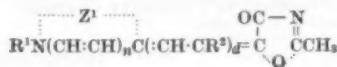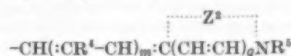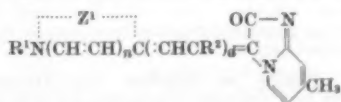$$R^1N(CH\!:\!CH)_nC(:CH\!\cdot\!CR^2)_d\!=\!C \overset{OC-N}{\underset{Q}{\big|}} CCH_3$$

(R¹ = subst. or unsubst. Alk, aralkyl or allyl; R² = H, Alk or Ar; Q = O, S or CONR³ (R³ = Alk or Ar); Z¹ = atoms to complete a 5- or 6-membered ring; $n = 0$ when Z forms a 5-membered ring and 0 or 1 when the ring is 6-membered; $d = 0$ or 1 or, when R² = H, 2) will react with a second molecule of one of the intermediates to yield deeply coloured dyes of the above formula in which the CH₃ group is replaced by

$$-CH(:CR^4\!\cdot\!CH)_m:C(CH\!\cdot\!CH)_qNR^5$$

(R⁴ = H, Alk or Ar; Z² = atoms to complete a 5- or 6-membered ring; $m = 0$ or 1 or, when R⁴ = H, 2; $q = 0$ when Z² forms a 5-membered ring and 0 or 1 when Z² forms a 6-membered ring).

                              BP 869,532 (U.S.A. 30 Aug 1956)

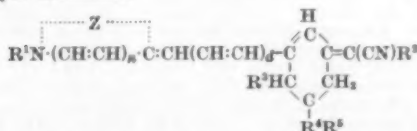 The same principle applies to dyes of formula

$$R^1N(CH\!\cdot\!CH)_nC(:CHCR^2)_d\!=\!C \overset{OC-N}{\big|} $$

(structure with N-CH₃)

                                                    C.O.C.

Methine and Polymethine Dyes

Kodak   BP 868,798 (U.S.A. 21 May 1956)

  Dyes of formula

$$R^1N\!\cdot\!(CH\!:\!CH)_n\!\cdot\!C\!:\!CH(CH\!:\!CH)_d\!=\!C \quad C\!=\!C(CN)R^2$$

(structure with H-C, R³HC, CH₂, C, R⁴R⁵)

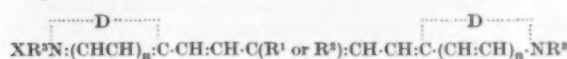(R¹ = Alk or Ar; n and $d = 0$ or 1; R² = cyano or carbalkoxyl; R³ = H or carbalkoxyl of 2 or 3 C; R⁴ and R⁵ = H or Alk; Z = atoms to complete a 5- or 6-membered ring), e.g. 2-[(3-dicyanomethylene-5,5-dimethyl-1-cyclohexen-1-yl)-methylene]-3-methylthiazolidine, have photosensitising properties.   C.O.C.

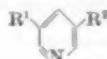## Dicarbocyanine Dyes

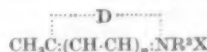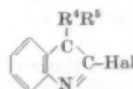Ilford   BP 867,403 (29 Apr 1958)

  Dyes of formula

$$XR^3N\!:\!(CHCH)_n\!\cdot\!C\!:\!CH\!:\!CH\!\cdot\!C(R^1\text{ or }R^2)\!:\!CH\!\cdot\!CH\!:\!C\!\cdot\!(CH\!:\!CH)_n\!\cdot\!NR^3$$

(R¹ and R² = H or Alk; R³ = Alk, aralkyl or Ar; D = atoms to complete a 5- or 6-membered ring; X = anion; $n = 0$ or 1) are obtained by treating a compound of formula

(structure: R¹ ring R² with N)

with one of formula

$$CH_3C\!:\!(CH\!\cdot\!CH)_n\!:\!NR^3X$$

in presence of one of formula

(structure: R⁴R⁵ ring with N, C-Hal)

(R⁴ and R⁵ = Alk; Hal = Cl or Br). Thus the bluish green bis-(3-methyl-2-benzothiazole)pentamethinecyanine iodide is obtained by adding 2-chloro-3,3-dimethyl-indolenine to 2,3-dimethylbenzothiazolium toluene-p-sulphonate suspended in anhydrous pyridine at room temperature and then heating for 150 min in a steam bath.   C.O.C.

Thermoplastic Resin Pigments and Compositions

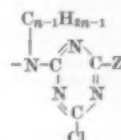Switzer Bros.   BP 869,801 (U.S.A. 11 June 1958)

  The above are obtained by dispersing dyes in finely divided thermoplastic resinous condensates of at least one aryl monosulphonamide having two reactive H on the amino N, and of a diamino-1,3,5-triazine or a C-Alk, Ar or Aralk derivative, and HCHO or an alkanol-modified methylol derivative of the aminotriazine. They have improved light fastness and colour brightness, enabling thinner films to be used to obtain optimum effects, and they can be ground to fine particle size without loss of strength. They are useful in coating compositions, especially printing inks, and in fluorescent dye compositions. Thus a mixture of Brilliant Yellow 6G Base (C.I. Solvent Yellow 44) and Rhodamine 6GDN Extra (C.I. Basic Red 1) is incorporated into the condensate of o- and p-toluene sulphonamide, paraformaldehyde and 2-phenyl-4,6-di-amino-1,3,5-triazine to give an orange-yellow colour of strong daylight fluorescence.   A.T.P.

Coloured Derivatives of High-molecular-weight Polyhydroxylated Compounds

Ciba   BP 871,087 (Switzerland 23 Sept 1957)

  High-mol.-wt. polyhydroxylated compounds, e.g. polyvinyl alcohol, pectins and especially cellulose, are dyed with a water-soluble reactive dye, especially one containing the group

$$-N\text{-}C \overset{C_{n-1}H_{2n-1}}{\underset{N}{\big|}} \quad \begin{matrix} N \\ C\text{-}Z \\ N \\ C \\ Cl \end{matrix}$$

(Z = subst. or unsubst. NH₂, OH, SH or Cl; $n = 1$–4), the dyed material being esterified, etherified, or acetalised to give highly stable coloured compounds, e.g. cellulose triacetates, which are useful as pigments and mass-coloured cellulose esters and, when partially hydrolysed to acetone-soluble acetates, as dyes for plastic compositions and cellulose acetate. Thus, the red dye (2)

(structure: HO, NH, N ring with C-Cl, C, N, Cl, -N:N-, SO₂H, HO₃S, SO₃H)

is dissolved in cold water (750) and cellulose powder (100) added during 30 min. NaCl (35) is added and, after 10 min, calcined Na₂CO₃ (2·5). After stirring for 90 min at room temperature, the dyed cellulose powder is filtered, soaped for 15 min at 90°C, and the dry dyed powder (10) pasted with N perchloric acid (50), filtered after 5 min and pressed to a wt. of 16 before being added

Colour Couplers

Kodak   *BP* 868,937 (U.S.A. 6 Sept 1956)

Colour couplers which are stable to heat and light in photographic emulsions and which yield dyes whose secondary light absorptions are low are of formula
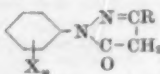
(X = Hal; n > 1; R = Alk of 11–17 C) e.g. 1-(2,4,6-trichlorophenyl)-3-pentadecyl-5-pyrazolone.   C.O.C.

Water-soluble Non-diffusing Dye Components for Colour Photography

Gasparcolor and G. B. Linden

USP 2,943,934 (8 May 1951)

Compounds of formula HNR^1R^2, H_2NR^3 and HOR^1 (R^1 = functional group rendering the compound capable of forming dyes; R^2 = H, Alk or Ar; R^3 = aromatic radical having an available coupling function), e.g. benzoylacet-(p-amino)anilide, 1-naphthylamine and 1,5-dihydroxynaphthalene, are rendered non-diffusing by treating them with compounds of formula $(-A(SO_2Cl)-)_n$ (A = organic radical; n > 2) e.g. chlorosulphonated polystyrene, to yield compounds of formula $(-A(SO_2Y)-)_n$, where at least some of the Y's = NR^1R^2, OR^1 or NHR^3, and some = Cl and after hydrolysis = an –O– cation group.   C.O.C.

Blue Metallisable Monoazo Dyes for Wool, etc.

FBy   *BP* 869,842 (Germany 5 Apr 1958)

Monoazo compounds

(Ac = acyl radical of carboxylic acid; X = OH or group converted to OH during metallisation; Alk is of 1–4 C) similar to those of *BP* 755,853 (J.S.D.C., **72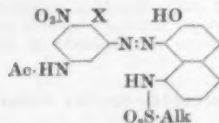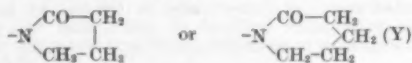** (1956) 545) are converted to Co or Cr complexes in substance or on the fibre to give blues. Thus 4-acetylamino-2-amino-4-nitrophenol is diazotised and coupled with 1-methyl-sulphonylamino-7-naphthol to give a blue monoazo metachrome dye which may be converted into the reddish blue 1 : 2 cobalt-complex.   E.S.

Metal(Chromium and Cobalt)-complex Monoazo Heterocyclic Dyes for Wool, etc.

S   *BP* 869,031 (Switzerland 20 July 1956)

The title dyes are neutral-dyeing 1 : 2 metal complexes of monoazo compounds, similar to those of *BP* 835,470 (J.S.D.C., **76** (1960) 512) and contain 1 or 2 substituents

Thus diazotised 2-amino-4-nitrophenol is coupled in presence of NaOH and Na_2CO_3 with 1-Y-7-naphthol, and the monoazo compound so formed is heated in formamide with chrome alum to give the Cr-complex which dyes wool, nylon, and leather black.   E.S.

Monoazo Disperse Dyes for Polyester Fibres

BASF   *BP* 870,677 (Germany 11 May 1957)

The title dyes are prepared by coupling diazotised p-nitroaniline and suitable derivatives thereof with tertiary amino derivatives of N-β-cyanoethylaniline, e.g. N-methyl-N-β-cyanoethyl-m-toluidine, the dyes being free from COOH or SO_3H groups, but optionally containing substituents, e.g. Alk, Hal, OAlk, $SO_2·NH_2$, CO·NH₂, etc. Thus diazotised p-nitroaniline coupled with N-butyl-N-β-cyanoethylaniline gives an orange.   E.S.
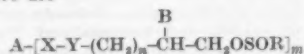
Azoic Dyes Containing Silicon

Union Carbide Corpn.   *BP* 870,467 (U.S.A. 29 Apr 1957)

The dyes of *USP* 2,927,829 (J.S.D.C., **77** (June 1961) 260) are applied to cotton by the azoic method. Thus cotton impregnated with β-naphthol becomes bright red when immersed in a solution of diazotised β-(aminophenyl)-ethyltriethoxysilane.   E.S.
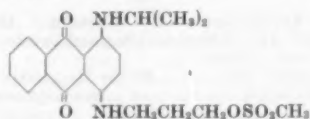
Anthraquinone Disperse Dyes

ICI   *BP* 870,948 (29 Oct 1958)

The above are

$$B$$
$$A-[X-Y-(CH_2)_n-CH-CH_2OSOR]_m$$

(A = subst. or unsubst. anthraquinone; B = H or OH; X = direct link, alkylamino or arylamino; Y = bridging atom or group; R = Alk, phenyl, tolyl or chlorophenyl; m = 1–2; n = 0–2, $n \neq$ 0 when B = OH, and A, X, Y, and R are free from SO_3H and COOH). They are prepared by any of the known methods and dye synthetic-polymer fibres, especially polyamides and modified polyacrylonitrile, bright colours of excellent fastness to light and washing. Thus, 1-isopropylamino-4-γ-hydroxypropyl-aminoanthraquinone (5·1) and methane sulphonyl chloride (2·25) are reacted at 0–5°C for 3 h in pyridine (75). Water (750) is added and the blue dye

filtered. It has excellent fastness to light and washing on nylon. Similarly a bright greenish blue dye is obtained from p-toluenesulphonyl chloride and 1-methylamino-4 p-(βγ-dihydroxypropoxy)anilinoanthraquinone.   A.T.P.

Quinophthalones— Yellow Disperse Dyes for Polyethylene Terephthalate

FH   *BP* 870,680 (Germany 25 Sept 1957)

The condensates of aromatic *ortho-* or *peri*-dicarboxylic anhydrides with subst. or unsubst. 2-methylpyridines or 2-methylquinolines are greenish yellow to reddish yellow dyes for polyesters (or, after sulphonation, acid dyes) and have good fastness. Thus, 4-fluorophthalic anhydride is condensed with quinaldine to give a clear greenish yellow disperse dye.   A.T.P.

2,2′-Difluoroacedianthrone— Brown Vat Dye

Ciba   *BP* 870,143 (Switzerland 4 Oct 1956)

This dye is obtained by cyclising bis-(2′-fluoro-9′-anthronylidene-10′)-ethane or -ethylene. It is faster to light than the 2,2′-dichloroanalogue.   A.T.P.

Methine Dyes

FBy   *BP* 869,794 (Germany 23 May 1957)

Methine dyes for mordanted cotton, leather, cellulose acetate, polyamides, polyurethane, and polyacrylonitrile fibres are

(R^1, R^4, R^5, and R^6 = Alk; R^2 = H or Alk; R^3 = H, Alk or Ar; X = anion, any of benzene nuclei may be substituted). They are obtained by condensing 7-alkylindole-3-aldehydes in acid solution with 1,3,3-trialkyl-2-methylene indolines, or by condensing 7-alkylindoles with 1,3,3-trialkylindoline-2-methylene-ω-aldehydes or by reacting 7-alkylindoles and 1,3,3-trialkyl-2-methyleneindolines with a dialkyl, diaryl, or alkylaryl formamide and halides of P or H_3PO_4. Thus, the condensate of 1,3,3-trimethyl-2-methyleneindoline-ω-aldehyde and 2-methyl-7-ethylindole dyes polyacrylonitrile brilliant orange.

A.T.P.

Red to Brownish Red Thiazine Dyes

Gevaert Photo-Producten   *BP* 869,138 (11 July 1958)

Condensing a phenothiazine aldehyde with a compound containing a reactive methylene or methyl group yields thiazine dyes specially useful for photographic filters or antihalation layers and which are permanently bleached when the exposed material is developed and fixed. Thus 3 - formyl - 10 - methylphenothiazine treated with 1-(p-sulphophenyl)-3-heptadecyl-5-pyrazolone yields the magenta

$$H_3C(CH_2)_{16}-C-C=CH-\ldots$$

(structure with S, N, CO, CH₂, SO₃H)

C.O.C.

Photosensitising Dyes

Ilford   *BP* 867,592 (19 June 1958)

Dyes of formula

(structure)

($n = 0$ or 1; D = atoms to complete a 5- or 6-membered ring; Z = one or more Alk, aralkyl, Ar, OH, alkoxy, NH_2, or substituted amino groups or H or Hal), e.g. the yellow 3-(benzothiazolyl-2)-7-hydroxycoumarin, have photosensitising properties.   C.O.C.

Merocyanine Dyes and Undissociated Cyanine Dyes

Kodak   *BP* 869,521 (U.S.A. 30 Aug 1956)

The methyl group of dyes of formula

(structure)

$$R^1N(CH:CH)_nC(:CH \cdot CR^2)_d=C \quad CCH_3$$

(R^1 = subst. or unsubst. Alk, aralkyl or allyl; R^2 = H, Alk or Ar; Q = O, S or $CONR^3$ (R^3 = Alk or Ar); Z^1 = atoms to complete a 5- or 6-membered ring; $n = 0$ when Z forms a 5-membered ring and 0 or 1 when the ring is 6-membered; $d = 0$ or 1 or, when $R^2 = H$, 2) will react with a second molecule of one of the intermediates to yield deeply coloured dyes of the above formula in which the CH_3 group is replaced by

$$-CH(:CR^4-CH)_m:C(CH:CH)_qNR^5$$

(R^4 = H, Alk or Ar; Z^2 = atoms to complete a 5- or 6-membered ring; $m = 0$ or 1 or, when $R^4 = H$, 2; $q = 0$ when Z^2 forms a 5-membered ring and 0 or 1 when Z^2 forms a 6-membered ring).

BP 869,532 (U.S.A. 30 Aug 1956)

The same principle applies to dyes of formula

(structure)

$$R^1N(CH:CH)_nC(:CHCR^2)_d=C$$

C.O.C.

Methine and Polymethine Dyes

Kodak   *BP* 868,798 (U.S.A. 21 May 1956)

Dyes of formula

(structure)

$$R^1N \cdot (CH:CH)_n \cdot C:CH(CH:CH)_d-C \quad C=C(CN)R^2$$
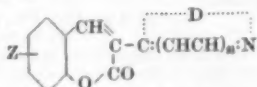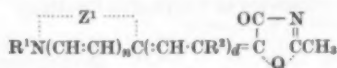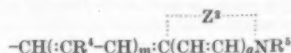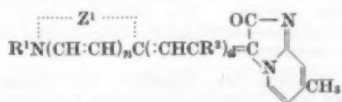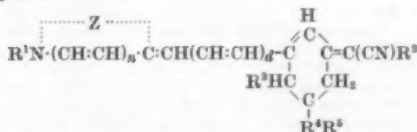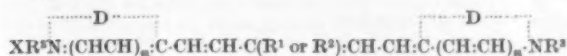
(R^1 = Alk or Ar; n and $d = 0$ or 1; R^2 = cyano or carbalkoxyl; R^3 = H or carbalkoxyl of 2 or 3 C; R^4 and R^5 = H or Alk; Z = atoms to complete a 5- or 6-membered ring), e.g. 2-[(3-dicyanomethylene-5,5-dimethyl-1-cyclo-hexen-1-yl)-methylene]-3-methylthiazolidine, have photosensitising properties.   C.O.C.

Dicarbocyanine Dyes

Ilford   *BP* 867,403 (29 Apr 1958)

Dyes of formula

$$XR^3N:(CHCH)_n \cdot C \cdot CH:CH \cdot C(R^1 \text{ or } R^2):CH \cdot CH:C \cdot (CH:CH)_n \cdot NR^3$$

(R^1 and R^2 = H or Alk; R^3 = Alk, aralkyl or Ar; D = atoms to complete a 5- or 6-membered ring; X = anion; $n = 0$ or 1) are obtained by treating a compound of formula

(structure)

$$R^1 \quad R^2$$

with one of formula

$$CH_3C:(CH \cdot CH)_n:NR^3X$$

in presence of one of formula

$$R^4R^5$$
(structure) C-Hal

(R^4 and R^5 = Alk; Hal = Cl or Br). Thus the bluish green bis-(3-methyl-2-benzothiazole)pentamethinecyanine iodide is obtained by adding 2-chloro-3,3-dimethyl-indolenine to 2,3-dimethylbenzothiazolium toluene-*p*-sulphonate suspended in anhydrous pyridine at room temperature and then heating for 150 min in a steam bath.   C.O.C.

Thermoplastic Resin Pigments and Compositions

Switzer Bros.   *BP* 869,801 (U.S.A. 11 June 1958)

The above are obtained by dispersing dyes in finely divided thermoplastic resinous condensates of at least one aryl monosulphonamide having two reactive H on the amino N, and of a diamino-1,3,5-triazine or a *C*-Alk, Ar or Aralk derivative, and HCHO or an alkanol-modified methylol derivative of the aminotriazine. They have improved light fastness and colour brightness, enabling thinner films to be used to obtain optimum effects, and they can be ground to fine particle size without loss of strength. They are useful in coating compositions, especially printing inks, and in fluorescent dye compositions. Thus a mixture of Brilliant Yellow 6G Base (C.I. Solvent Yellow 44) and Rhodamine 6GDN Extra (C.I. Basic Red 1) is incorporated into the condensate of *o*- and *p*-toluene sulphonamide, paraformaldehyde and 2-phenyl-4,6-di-amino-1,3,5-triazine to give an orange-yellow colour of strong daylight fluorescence.   A.T.P.

Coloured Derivatives of High-molecular-weight Polyhydroxylated Compounds

Ciba   *BP* 871,087 (Switzerland 23 Sept 1957)

High-mol.-wt. polyhydroxylated compounds, e.g. poly-vinyl alcohol, pectins and especially cellulose, are dyed with a water-soluble reactive dye, especially one containing the group

$$C_{n-1}H_{2n-1}$$
(structure with N-C, C-Z, N, N, C, Cl)

(Z = subst. or unsubst. NH_2, OH, SH or Cl; $n = 1$–4), the dyed material being esterified, etherified, or acetalised to give highly stable coloured compounds, e.g. cellulose triacetates, which are useful as pigments and mass-coloured cellulose esters and, when partially hydrolysed to acetone-soluble acetates, as dyes for plastic compositions and cellulose acetate. Thus, the red dye (2)

(structure with HO, NH, N, C, C-Cl, N, N, C, Cl, N:N, SO₃H, HO₃S, SO₃H)

is dissolved in cold water (750) and cellulose powder (100) added during 30 min. NaCl (35) is added and, after 10 min, calcined Na_2CO_3 (2·5). After stirring for 90 min at room temperature, the dyed cellulose powder is filtered, soaped for 15 min at 90°C, and the dry dyed powder (10) pasted with N perchloric acid (50), filtered after 5 min and pressed to a wt. of 16 before being added

to benzene (320) at 0°C. Acetic anhydride (160) is added and after 150 min the product, a red cellulose triacetate, is filtered off and washed.   A.T.P.

Stabilised Dyes for Polyvinyl Acetal Resins
DuP   *BP* 869,986 (U.S.A. 29 Apr 1957)
Stabilised dyes for use in polyvinyl acetal resins comprise a dye and a dialkyl dithiocarbamate chelate of Ni, Co, Zn, Mn or Cr, or an alkyl xanthate, 2-mercaptobenzothiazole or salicylaldehyde ethylenedi-imine chelate of Ni or Co (wt. chelate to dye $\ll 1:10$). The dye stabiliser inhibits the effect of sunlight without impairing the colouring properties of the dye. The compounds are especially useful for interlayers of automotive safety-glass and of decorated laminated articles and for sheets for coloured windows.   A.T.P.

Red Food Dye based on Animal Blood Haemoglobin
Griffith Laboratories   *USP* 2,938,800 (3 Oct 1958)
Blood or "wet pigment solids", i.e. the product obtained by centrifuging blood, is vacuum dried to a 12–15% water content. It is then prevented from becoming brown (caused by rapid attack by enzymes or micro-organisms) by adding to it a water-sharing edible material, e.g. dextrose, sucrose and dry syrup solids, together with a preservative, e.g. propylene glycol. The product is used as a red food dye particularly for meat, fish pastes and vegetable-base sausage.   C.O.C.

Carotenoids by Cultivation of Algae
Grain Processing Corpn.   *USP* 2,949,700 (21 July 1958)
High yields of β-carotene (C.I. 75130) and xanthophyll are obtained by cultivating algae in an aqueous organic medium containing urea as a supplementary source of fixed nitrogen.   C.O.C.

Non-leafing Aluminium Flake Pastes (C.I. Pigment Metal 1)
American-Marietta Co.   *BP* 869,091 (U.S.A. 26 June 1957)
Aluminium is mechanically reduced to flakes in presence of a lubricant and during the process is treated with an aqueous solution at pH 1·5–11·0 containing phosphoric acid or an NH_4, alkali-metal, alkaline-earth metal, Zn, or Mg phosphate. The amount of solution should be such that there is present $\ll 0.7\%$ by wt. of the aluminium of available PO_4 ion. The product is readily miscible with aqueous coating compositions.   C.O.C.

Alumina–Silica Pigments
Columbia-Southern Chemical Corpn.
                                USP 2,949,379 (18 Mar 1957)
Ca or other alkaline-earth silicate is treated with enough Al sulphate to react with 30–70% of the silicate. The precipitate is filtered off, dried, and then treated with an aqueous solution of sufficient of an Al salt to react with $\ll 50\%$, and preferably all, of the CaO present. The product, of which $\ll 90\%$ by weight consists of 0·5–7·0 μ flocs, is an excellent opacifying pigment for paper, and the last stage of its manufacture may, if desired, be carried out in the pulp slurry.   C.O.C.

V—PAINTS; ENAMELS; INKS

Basic Pigments
A. G. Walker   *Off. Dig. Fed. Soc. Paint Technol.*,
                                      33 (Apr 1961) 481–491
Basic pigments are defined as those which, by virtue of their chemical composition, can react with acidic components of the paint medium to produce "soaps". These "soaps" reduce the corrosive action of water with which they come into contact. The properties and use of the following are discussed: basic lead sulphate, basic lead chromate, basic magnesium chromate, red lead, and calcium plumbate.   J.W.D.

Soluble Pigments
H. F. Clay   *Off. Dig. Fed. Soc. Paint Technol.*,
                                      33 (Apr 1961) 492–505
Ideally the ratio of sol. impurities in chromate pigments used in corrosion-inhibitive coatings should be low, under which conditions perfect anodic inhibition is produced initially, followed by the development of excellent cathodic inhibition. If the ratio of impurities to $CrO_4{}^{2-}$ ions is high the development of anodic inhibition is impaired and some attack will take place. In the case of zinc potassium chromate this attack will be minimised or arrested by the rapid development of cathodic inhibition. Best results are obtained with chromate pigments when Cl^- and $SO_4{}^{2-}$ are virtually absent and there is no justification for relaxing the limits at present set upon such impurities.   J.W.D.

Metallic Pigments in Protective Coatings
A. Pass and J. F. Meason
                                Off. Dig. Fed. Soc. Paint Technol.,
                                      33 (Apr 1961) 506–524
Metallic aluminium affords a mainly physical protection against corrosion by excluding corrosive elements from the substrate, while at the same time imparting high electrical resistance to the film. Lead not only produces films of high integrity as with Al, but also produces inhibitive films by lead–soap decomposition reactions; in certain cases it gives some measure of cathodic protection. Zinc functions positively, firstly by cathodic protection, which is gradually replaced by an anodic passivation reaction which protects the Zn itself as well as the metal substrate, and finally leads to an extremely high order of barrier protection.
                                                              J.W.D.

Pigment Dispersing Aids
F. J. Prescott
                    Paint Varnish Production, **50** (12) (1960) 31–35, 91–92
                              Chem. Abs., **55** (29 May 1961) 10915
N-methyl-2-pyrrolidinone and *N*-vinyl-2-pyrrolidinone are effective dispersing aids for a variety of organic and inorganic pigments. Fineness, tinctorial strength, gloss, and reduced flocculation resulted from use of some of them in various vehicles. The solubility and dispersing properties of *N*-methyl-2-pyrrolidone suggest its use as a universal tint carrier.   C.O.C.

Silicone Resins as Binders for Heat-resistant Varnishes and Suitable Pigments for Use therein
W. Kravs
                    Elektrotech. u. Maschinenbau, **74** (1957) 349–355
                              Chem. Abs., **55** (29 May 1961) 10916
Methyl and methylphenyl silicone resins, especially the latter, are the only silicone resins compatible with pigments. Pigments suitable for use with these resins are TiO_2 (C.I. Pigment White 6), Cadmopur and Cadmofix, Chromium Oxide Green (C.I. Pigment Green 17), Iron Oxide Green, Manganese Blue, mixed pigments of Cr, Co, Ni and Fe oxides, Al bronze (cf. C.I. 77000), and Zn powder (C.I. Pigment Metal 6). Irisol Fast Yellow GRE, Irisol Fast Violet BBN, and Irisol Fast Blue BL are the only organic pigments stable at the temperatures at which silicone resins are applied. Details of the manufacture, application, and properties of varnishes based on these resins and pigments are given.   C.O.C.

Hydrated Aluminium Silicates of Large Particle Size as Flatting Agents
G. P. Larson
                    Paint Varnish Production, **50** (12) (1960) 39–44, 90
                              Chem. Abs., **55** (29 May 1961) 10915
This type of kaolinite makes available for the first time a low-oil-absorption flatting agent for control of gloss and sheen. Use of such an agent also imparts improved

enamel hold-out, lower viscosity, and better abrasion resistance to alkyd and emulsion paint systems.   C.O.C.

Method for Determining Hiding Power and its Application as an Aid to Paint Formulation

P. I. Adams   *J. Oil & Col. Chem. Assocn.,*
                                                 44 (May 1961) 295–307

Contrast hiding ratio (CHR) values for different thicknesses of dry paint are found by measuring the reflectance of films applied to black and white printed tinplate by spraying. From knowledge of the formulation and the dry paint wt., vol. of pigment present per unit area in the paint films are calculated. A method of plotting the results is described which, when applied to a series of paints of differing concn. of a given pigment in a given medium, shows the variation of CHR with both pigmentation and vol. of pigment per unit area. Critical pigment vol. concn. may be deduced from the hiding power graphs. Calculations based on paint wt., non-volatile content, and density measurements permit study of the variation in CHR with pigmentation and wet-film thickness.   Interpolation and extrapolation of results are discussed with a view to the possible expression of results in terms of spreading rate at given opacity levels.   J.W.D.

Catalyst for Aminoplast Condensates

Rohm & Haas Co.   BP 867,806 (U.S.A. 24 Oct 1956)
The monosalt of maleic acid with triethylamine is an excellent catalyst for use with aminoplast condensates giving comparatively rapid curing. This renders them suitable for use in alkyd aminoplast coating compositions, where their presence results in increased gloss and adhesion.   C.O.C.

Basic Dye Salts of Polysulphonated Phthalocyanines as Colorants for Ballpoint Pen Inks

DuP   USP 2,950,285 (30 June 1958)
An ink of good strength and free from caking or polymerisation troubles is obtained by incorporating into a glycollic solvent the dye salt obtained by mixing a solution of Victoria Pure Blue BO (C.I. Basic Blue 7) with one of a metal or metal-free phthalocyanine polysulphone containing on average 2–4 sulpho groups per molecule. Writing obtained with this ink is of superior fastness to light and rubbing.

                             USP 2,950,286 (30 June 1958)
The solubility of basic dye salts of sulphonated phthalocyanine in glycollic solvents depends on the degree of sulphonation, at least 2 sulpho groups per mol. being necessary. Reaction of salt formation between such phthalocyanine polysulphonic acids can be carried out in two distinct manners depending upon whether a granular solid or non-filterable tarry mass is desired. Details of both methods are given.   C.O.C.

Readily Removable Marking Composition

National Cash Register Co. USP 2,950,256 (4 Mar 1957)
An ink is made up of a light-active sublimable substance in a liquid which can be readily evaporated to leave the sublimable substance uncovered, so that it may sublime, e.g. anthracene (400 g), naphthacene (4), ethyl hexane-diol (10 l.), and xylene (4 l.) yields markings which fluoresce in ultraviolet radiation and are readily removed by exposure for > 2 s at 300°F.   C.O.C.

Daylight Fluorescent Compositions

Lawter Chemicals   BP 870,357 (U.S.A. 11 June 1958)
The compositions (inks, pigments, coating compositions) comprise a daylight fluorescent colorant, e.g. Xylene Red B (C.I. Acid Red 52), Rhodamine BX (C.I. Basic Violet 10), Brilliant Sulpho Flavine FFA (C.I. Acid Yellow 7), and the co-condensate of benzoguanidine (I) and HCHO with an arylsulphonamide (II) and HCHO (preferably 0·3–0·6 I per unit II). The compositions have improved thermal stability, light-fastness, and wet-out characteristics.   A.T.P.

Heat-reactive Compositions containing a Polyamide and Trimethylphenol

Union Carbide Corpn.   USP 2,946,759 (23 June 1958)
A homogeneous mixture of trimethylolphenol (> 98 parts) and a polyaminoamide (< 2) cures rapidly to the infusible state at low temperatures. It yields coatings which adhere tenaciously to the substrate and which are flexible and impervious to chemicals and solvents.

                                                 C.O.C.

Corrosion-resistant Coating Comprising a Furfuryl Alcohol Resin, Cellulose, Filler, and Catalyst

C. S. Price   USP 2,948,639 (15 May 1958)
A sprayable composition yielding coatings which are resistant to acids, alkalis, and ultraviolet radiation is obtained by mixing a furfuryl alcohol resin precondensate, filler, cellulose, and solvent. An acid or acid-forming polymerisation catalyst is added to the homogeneous mixture before spraying starts so that curing begins after spraying has been completed.

                                                 C.O.C.

Thermoplastic Resin Pigments and Compositions (IV p. 457)
Coloured Derivatives of High-molecular-weight Polyhydroxylated Compounds (IV p. 457)

VI— FIBRES; YARNS; FABRICS

Degradation of Fibres by Dyes

F. Dorr   *Chimia,* **15** (Jan 1961) 63–67
Description of relevant electronic configurations, and their effects, which lead to the absence or presence of fibre-degrading properties in anthraquinonoid vat dyes. Thus, an oxygen radical produces a powerful dehydrogenating effect and the triplet state of π-electrons transmits oxygen to the fibre. Pyranthrone, Flavanthrone, Indanthrene Blue RS, Indanthrene Yellow 4GF, Algol Yellow GC, and tetrabenzpyrenequinone are all fully discussed.
                                                 A.T.P.

Status of Non-woven Fabrics in the Textile Trade

H. E. Shearer
              Amer. Dyestuff Rep., **50** (17 Apr 1961) P292–P298
A brief review of the position of non-woven fabrics is given. Recent advances in web-forming equipment, both wet and dry, and in bonding equipment are discussed. The use is outlined of thermoplastic bonding fibres, self-bonding rayon, and chemical binders.   S.B.D.

Change in Properties of Acid-damaged Cotton Fabric

A. Parisot
          Bull. Inst. Text. France, (93) (Mar–Apr 1961) 7–18
Cotton fabric was treated at 40°C for 30 min with different concentrations of HCl, H_2SO_4, and H_3PO_4. The wet (w) and dry (d) bursting strengths and the D.P. of the cellulose were measured on the treated fabric. Rate of hydrolysis was found to depend only on the pH of the solution, the nature of the acid having little effect. The rate was slow at pH $>$ 2. w and d decreased as hydrolysis proceeded. The difference w–d also decreased, becoming zero at D.P. values in the range 550–750, whatever the conditions of hydrolysis. In this respect, acid hydrolysis affects the mechanical properties of the fabric in the same way as oxidative degradation by hypochlorite and perborate solutions.   J.C.F.

Aminisation of Cotton: Cost Analysis Application

S. P. Kolton, K. M. Decossas, G. L. Drake, E. F. Pollard, and E. L. Patton
              Amer. Dyestuff Rep., **50** (3 Apr 1961) 241–244
Cotton in which aminoethyl groups are introduced (AM cotton), has excellent affinity for wool-type acid dyes, good ion-exchange capacity, and a high degree of reactivity. This reactivity lends itself to further chemical modification. Cost data are provided that are needed for considering the feasibility of producing AM cotton. Investment and processing costs are compared with those reported in an earlier article (see *Amer. Dyestuff Rep.,* **48**, No. 17 (1959) 37).   S.B.D.

Reaction between Urea and Cellulose

L. Segal and F. V. Eggerton
                   Text. Research J., **31** (May 1961) 460–471
Cellulose heated with an aqueous solution of urea becomes insoluble in cupriethylenediamine. In addition its infrared absorption spectrum is altered, its nitrogen content is unaffected by boiling water, it has changed dyeing properties and modified regain. There is no change in the cellulose X-ray diffraction pattern and all the original cellulose properties are restored by boiling in 2% aq. NaOH. The treatment does not affect the textile properties of cotton fabric. Biuret acts similarly to urea.
                                                 C.O.C.

Modes of Deformation of Cotton Fibres

M. S. Nigmankhodzhaeva and Kh. U. Usmanov

Khim. Khlopchatnika, Akad. Nauk Uzbek. S.S.R., Inst. Khim. Rastitel. Veshchestv., (1959) 25–39

Chem. Abs., **55** (29 May 1961) 10896

Differences in behaviour between wet and dry cotton and rayon fibres under applied stresses and when relaxed were examined and are discussed on the basis of the theory of elastic (ϵ_e), viscoelastic (ϵ_c) and plastic (ϵ_p) deformations. For cellulose fibres the total deformation is $\epsilon = \epsilon_e + \epsilon_c + \epsilon_p$. In completely dry fibres, ϵ_p is absent and ϵ_c increases slowly and linearly with the load. In moist fibres, ϵ increases with time on repeated alternate application and removal of load, while ϵ_p and ϵ_c decrease. The value of ϵ under such conditions is greater than that under single, continuous stress for both cotton and rayon. Apparently the effect of moisture on cellulose, in particular on cotton, is analogous to that of heat on polymers or plasticisers on plastics.   C.O.C.

Stabilisation of Viscose Rayon by Heat Treatment after an Ammoniacal Preswelling Bath

K. Kanamaru

J. Applied Polymer Sci., **4** (July–Aug 1960) 1–9

Viscose rayon, preswollen in an ammonium hydroxide bath of optimum concentration or pH range for the maximum swelling of the rayon, is heat-treated at a temperature just above the glass-transition temperature of regenerated cellulose. The improvement in dynamic modulus, breaking strength, and crystallinity (from X-ray measurements) is greater than that after preswelling in acid followed by heat treatment.   P.T.S.

Ethanolysis of Viscose Fibres

E. A. Abramova and O. Ya. Bufetchikova

Zhur. priklad. khim., **34** (Feb 1961) 416–423

Fibres prepared from viscose soln. differing in the degree of substitution of the xanthate and in the amount of $ZnSO_4$ in the coagulating bath (0–105 g/l.) have been submitted to ethanolysis and the kinetic data related to fibre fine structure. It is shown that the amount of amorphous fraction in the fibre increases with increase in $ZnSO_4$. Lowering of the degree of substitution of the xanthate results in reduced hydrolysability of the fibre.   G.J.K.

Sorption of Water Vapour by Hydrophilic High Polymers. III— Sorption Isotherms and Heat of Wetting of Cellulose Acetate

E. F. Nekryach and Z. A. Samchenko

Ukrain. khim. zhur., **26**, No. 6 (1960) 700–706

Cotton linters were subjected to various degrees of acetylation to give four samples A_1 to A_4, where A_4 was nearly pure cellulose triacetate. Heats of wetting at 20°C and sorption isotherms of these samples were measured. At low r.h. ($P/P_s = 0.03$) cotton linters absorb appreciably more moisture than do acetylated samples. At high r.h. ($0.03 < P/P_s < 0.9$) there is very little difference in absorption of cotton linters, A_1, A_2, and A_3. At all values of P/P_s absorption of moisture by A_4 is the lowest. In samples containing originally less than 0.25 g mol water/100 g fibre, the heat of wetting decreases in series cotton linters $> (A_1, A_2, A_3) > A_4$. If the original water content is greater than 0.25 g mol/100 g fibre, heats of wetting of cotton linters, A_1, A_2, and A_3 are similar, and greater than that of A_4. Differential thermodynamic functions (free energy, entropy, enthalpy) of the process of combination with water were determined experimentally for pure and acetylated celluloses. From the examination of entropy curves the energy involved in combination of a water molecule with a hydrophilic group of cellulose was determined. This energy decreases in order primary hydroxyl groups $>$ acetylated group $>$ secondary hydroxyl group.   L.S.L.

Influence of Three-dimensional Configuration on Reactivity and Stability of Proteins

C. B. Anfinsen   *J. Polymer Sci.*, **49** (Jan 1961) 31–49

A review of the effect of protein tertiary structure and quaternary structure on the ;reactivity of individual amino-acid side-chains and on the stability of the protein to attack on the main chain by enzyme, chemical, or heat treatment.   P.T.S.

Structural-mechanical Properties of Protein Fibres

N. V. Grigor'eva, V. A. Pchelin, and P. A. Rebinder

Doklady Akad. Nauk S.S.S.R., **137** (4) (Apr 1961) 889–892

Model gelatin fibres have been prepared in order to determine the effect of tanning upon the mechanical properties of protein fibres (in leather). The kinetics of deformation (extension) and of relaxation of tanned and crude gelatin fibres have been determined. The elasticity moduli of both kinds of gelatin fibres are practically the same (approx. 600 kg/mm²), but the values of residual deformation after relaxation are 2% for tanned fibres and 6% for crude ones. The effect of tanning is still more apparent in the case of moist fibres, where the respective values of residual deformation are 16 and 33%. The increase of temperature to 45°C during relaxation reduces the residual deformation of tanned fibres to 4% and that of crude fibres to 30%.   I.G.

Different Permeability of Cuticular Sheath in Normal and Guard Keratin Fibres

R. Z. de la Burde   *Nature*, **190** (29 Apr 1961) 420–422

The guard hair of the rabbit is considerably more resistant to the penetration of Acridine Orange (C.I. Basic Orange 14) and Aniline Black (C.I. Pigment Black 1) than is normal rabbit hair. The cuticular sheath of guard hair is responsible for the phenomenon— if the cuticle is damaged, uniform staining of the fibre occurs. The cuticular sheath is impermeable to Acridine Orange, regardless of the number of treatments, whereas its resistance to penetration gradually decreases in repeated treatments with Aniline Black, and it is probable that the latter modifies the keratin of the cuticle. The difference in behaviour between normal and guard hairs is attributed to differences in keratinisation of the cuticle.   J.C.F.

Nitrosation of Tyrosyl Residues— Effect on Properties of Wool— II

M. Leveau   *Bull. Inst. Text. France*, (93) (Mar–Apr 1961) 75–80

(For Part I see J.S.D.C., **77** (Mar 1961) 134.) Complete amino-acid analysis, by ion-exchange resin chromatography, of normal wool and wool treated with nitrous acid showed that nitrosation attacks almost all amino acids to a greater or less extent. Lysine, tyrosine, and phenylalanine are particularly affected. In the case of tyrosine, there is a large discrepancy between the chromatographic result and a direct Folin determination on the hydrolysate: this may be due to deamination of tyrosine in the peptide linkage, which destroys the amino but not the phenolic group.   J.C.F.

Electron-microscopic Investigation of Fibrillar Structure of Silk Fibres

M. Minagawa   *J. Soc. Textile Cellulose Ind. Japan*, **17** (Apr 1961) 335–340

Examination under the electron microscope of silk fibre (from which sericin has been removed) whilst it was undergoing treatment with hydrogen peroxide or caustic soda indicated that fibrils of diameter 400–600 Å were formed from a large number of fibrils of diameter 100–150 Å and $0.2–0.4\,\mu$ and width $0.6–0.8\,\mu$. During the process of fibre breakdown, fibrils of diameter $0.2–0.4\,\mu$ tend to develop into band-form fibrils of width $1.0–1.2\,\mu$. With the exception of wild silk and one group of domestic cocoon, the middle fibroin layer shows poor resistance to hydrogen peroxide and caustic soda compared to the inner and outer fibroin fibre layers.   I.G.L.

Effects of Phosphates and Silicates on Silk. VII— Effects of Condensed Phosphates on Silk in Tin Weighting

E. Omura, A. Hirata, and M. Kobayashi

J. Soc. Textile Cellulose Ind. Japan, **17** (Apr 1961) 351–353

Tin weighting, using stannic chloride, has been carried out on degummed raw silk and Habutae fabrics with the use of condensed sodium phosphates, phosphates ($Na_5P_3O_{10}$, $Na_4P_2O_7$, $(NaPO_3)_6$, NaH_2PO_4, Na_2HPO_4, Na_3PO_4, KH_2PO_4, and K_2HPO_4) and sodium metasilicate as assistants. It is concluded that— (1) The weight percentage increases as the number of treatments involving the above assistants increases. (2) The weight percentage increases when high concentrations of condensed phosphates and sodium metasilicate are employed, but falls

considerably when solutions of concentration 0·5–0·12% are used. However, at even lower concentrations, there is a further gradual increase in the weight percentage. (3) The effects of the potassium salts are less dependent on temperature and concentration than those of the sodium salts, and the weight percentage is relatively constant.

                                                                I.G.L.

New Fibres from Du Pont
M. M. Christie
          Amer. Dyestuff Rep., **50** (1 May 1961) P340–P342
    A description of the textile and dyeing properties of five new fibres. Orlon Type 72 and Sayelle, Antron nylon, and Dacron Type 62 are modifications of established products, while Lycra Spandex is a new fibre of modified polyurethane material.   P.B.S.

Hygienic Properties of Synthetic-fibre Fabric
I. A. Dimitrieva   *Tekstil. prom.*, No. 1 (Jan 1961) 53–55
    Steam permeability, hygroscopicity (absorption from humid air) and water absorption by capillary attraction of various synthetic-fibre fabrics have been measured. The influence of fabric structure (density, thickness) on these properties has been examined. Certain comparisons with natural fibres have also been attempted.   L.S.L.

Degradation and Cross-linking in Irradiated Polyamides and the Effect of Oxygen Diffusion
J. Zimmerman   *J. Polymer Sci.*, **46** (Sept 1960) 151–162
    Irradiation of nylon 6.6 with electrons of 2 meV causes cross-linking and chain rupture. The changes are followed by changes in relative viscosity of the polymer solution. Apparent rate of oxygen diffusion into the polymer is lowered because of combination with radicals formed during irradiation. The dependence of degradation on fibre thickness is related to the rate of oxygen diffusion.
                                                                P.T.S.

Photochemical Degradation of Nylon 6.6 Treated with Size Based on Acrylic Acid
P. Rochas and S. Pierret   *Bull. Inst. Text. France*,
                              (93) (Mar–Apr 1961) 81–86
    Sizes based on acrylic acid do not influence the photochemical degradation of nylon 6.6 yarns.   J.C.F.

Fine Structure of Polyamides. III— Effect of Liquid Baths on Structure and Properties of Spun Bristles and Fibres of Polycaprolactam
C. Ruscher, V. Gröbe, and H. Versäumer
        Faserforsch. und Textiltech., **12** (May 1961) 214–222
    Different mesomorphic and crystalline arrangements of molecules are found in spun polycaprolactam fibres and these are affected differently by thermal, chemical, and mechanical treatments. Spinning fibres into a tube filled with liquid causes orientation of molecules which depends on the temperature and affects the stretching energy. Conclusions as to practical spinning conditions are drawn.
                                                                W.R.M.

Effect of an Airborne Soil on the Photochemical Degradation of Nylon
M. A. Morris and B. W. Mitchell
                    Text. Research J., **31** (May 1961) 488
    Nylon treated with an airborne soil lost 22 and 24% more strength than clean nylon after 160 and 320 h, respectively, exposure in the Fade-Ometer. Corresponding difference in strength between soiled and clean yarns did not result from ageing or heating.   C.O.C.

Polyolefins in Textiles
W. Henstead   *J. Textile Inst.*, **52** (Apr 1961) P158–P168
    After a brief historical introduction the physical and chemical properties of low-density branched polyethylenes, high-density linear polyethylenes, and polypropylene are compared. The spinning of yarns and their properties and end-uses are discussed.   W.P.M.

Resistance of Fibres prepared from Mixtures of Polyvinyl Alcohol and Polyacrylic Acid to Water, Acid, and Alkali
K. Kawashima   *J. Soc. Textile Cellulose Ind. Japan*,
                              17 (Apr 1961) 330–334
    Fibres prepared from a mixture of polyvinyl alcohol and polyacrylic acid were heat-treated and formalised, and their resistance to water, acid, and alkali was investigated. The resistance of unformalised fibres to water increases (a) on heat-treatment and (b) as the polyacrylic acid content is increased. A fibre containing 25–30% polyacrylic acid, heat-treated at 220°C, exhibits fairly high resistance to

boiling water, comparable with that of ordinary vinylon. The effects of HCl (0·4 and 3·5 N), H_2SO_4 (3·5 N), Na_2CO_3 (0·4 N), and NaOH (0·4 and 3·5 N) have been studied. It is concluded that an increase in the polyacrylic acid content results in an increase in the resistance of the formalised fibre to hydrochloric acid, whereas the resistance to alkali decreases.   I.G.L.

Polyester Filaments and Fibres having a Metallic Lustre
DuP   USP 2,948,583 (4 Mar 1958)
    Polyester filament or film having an initial birefringence of $>$ 0·01, is drawn at its natural draw ratio at $<$ 80°C to $<$ 5 times its original length at a drawing speed of $<$ 0·75 and $>$ 50·0 yd/min. This gives the polyester a metallic lustre, high tenacity, and improved surface characteristics.   C.O.C.

Stabilising the Colour of Acrylonitrile Polymers
Chemstrand Corpn.   USP 2,946,760 (23 May 1956)
    Addition of an aldehyde and an inorganic acid of ionisation constant $> 1.86 \times 10^{-5}$, e.g. HCHO and H_2SO_4, stabilises a polyacrylonitrile solution against formation of colour on standing and also prevents colour formation when fibres etc. formed from such solutions are stored or heated.
                            USP 2,946,761–2 (1 Nov 1956)
    Aqueous solutions of polyacrylonitrile are stabilised against colour formation if 0·1–5·0% (on the wt. of the polymer) of a water-soluble inorganic bisulphite is present.
                                                                C.O.C.

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Current Desizing Techniques
J. A. D. Hall   *Dyer*, **125** (19 May 1961) 727–730
    Unremoved starch leads to increased consumption of chemicals and fixed residues in chlorite and peroxide bleaching, and to less permanent fixation in resin finishing. The bacterial desizing enzymes are preferred to malt and pancreatic types. Low enzyme strengths are suitable for batch processes; medium to high strengths are more suitable for continuous processes. An efficient roller-batching desizing method is described. A brief history of continuous desizing, viz. J box, heated cans and the recent heated drum and blanket reactor, is given.
                                                                R.A.P.

Control of Bleaching
A. Kling and J. Kurz
                    Textil-Rund., **16** (May 1961) 253–258
    The degree of whiteness of a fabric treated with a fluorescent brightening agent depends upon both reflected and fluorescent light. Separate measurement of these is necessary for the quantitative assessment of whiteness and this cannot be done visually. By illuminating the fabric with light free from u.v. radiation the operation of the fluorescent brightening agent is prevented and the true reflected light can be measured. The fluorescent light is obtained by difference from the apparent total reflected light. Measurement with a spectrophotometer is simplest, but a reflectance photometer can be used with an additional filter. The operation of each type of apparatus is described and an example is given.   P.B.S.

Use of Irradiated Potassium Chlorate as a Bleaching Agent
A. S. Baberkin, T. V. Bromberg, L. I. Kirkina, and L. I. Belen'kii
          Tekhnol. tekstil. prom., No. 1 (20) (1961) 103–105
    Potassium chlorite obtained from the decomp. of the chlorate by γ-rays from ^{60}Co has given very effective bleaches with less tendering of cellulose than occurs in the comparable hydrogen peroxide systems.   G.J.K.

Bleaching with Sodium Chlorite

J. F. Barton *Text. Manuf.*, **87** (May 1961) 205–207

Points dealt with include the type of machinery, open-width equipment, rope bleaching of cotton piece-goods, winch bleaching of knitted fabrics; bleaching of nylon and Terylene, bleaching efficiencies; and control of pH. S.B.D.

Variables in Hydrogen Peroxide Bleaching of Cotton

H. L. Martin

Canadian Textile J., **78** (12 May 1961) 42–44

The variables encountered in bleaching, their inter-relation, their relation to bleaching faults, and the influence of knowledge of them on the development of recent improved bleaching methods, are discussed. W.P.M.

Continuous Single-bath Scouring and Bleaching of Cotton Fabrics using Stabilised Hydrogen Peroxide

N. E. Fedorova and P. V. Morÿganov

Tekhnol. tekstil. prom., No. 2 (21) (1961) 96–103

The stabilising effects of $Na_5P_6O_{18}$ (I), Na_2SiO_3 (II), with and without $MgSO_4$; of I plus II; and of I plus II plus $MgSO_4$, on the decomp. of H_2O_2 in alkaline soln. (6% NaOH) at 98°C are investigated and the superiority of II plus $MgSO_4$ is established. A processing sequence in which desizing is immediately followed by bleaching in a sodium-silicate-stabilised H_2O_2 bath is described in detail. G.J.K.

Comparative Wool Scouring Studies. I— Yield and Regain Differences between Solvent and Aqueous Methods

V. A. Williams *Text. Research J.*, **31** (May 1961) 472–477

In laboratory trials, solvent scouring gave a higher yield than aqueous alkaline scouring, 0·5–1·4% more clean dry fibre being obtained in solvent degreasing than in traditional soap–soda scouring. The difference in yield is caused by some of the oxidised protein material in the tips of the fibres being dissolved by hot alkaline scouring liquors. Wool cleansed by solvent or a neutral detergent has a lower regain than that from alkaline scouring. C.O.C.

Mercerisation of Piece Goods on Chain or Chainless Machines

E. Pierling *Melliand Textilber.*, **42** (Apr 1961) 451–454

A new chainless mercerising machine is described and the following advantages are claimed. The tension of the goods is automatically controlled by double compensators. The passage of the fabrics through the entire plant is synchronised. A novel vibration device ensures highly efficient removal of caustic soda. Much improved washing effects are obtained with the Turbotex machine. When the fabrics pass through a compensator, a certain pre-determined amount of warp tension is imparted to them, and tension is further increased on passing through the second and third compensators. Wetting-out is facilitated by a special arrangement of rollers which force the caustic soda through the goods. Experiments in a mill with both chain and chainless machines showed that goods treated on a chainless machine had 15–20% (measured) more lustre than those treated on the chain machine. W.M.

Continuous Soaking of Loose Fibres with Detergent Liquid

A/B Separator *BP* 867,451 (11 Dec 1958)

Loose fibres, e.g. raw wool, are led into detergent liquor kept in continuous agitation by injection of a jet of the liquor below the liquor level. The jet is injected towards an outlet at the bottom of the opposite wall of the vessel so that the fibrous suspension formed is discharged through that outlet. C.O.C.

VIII— DYEING

Package Dyeing

R. Weber *S.V.F. Fachorgan*, **16** (Mar 1961) 178–193

Early records show that the first liquor-circulating machine was patented in 1858, and 40 years later a patent was granted for a machine operating at above 100°C. During the last 50 years advances have been made in the construction materials and design of liquor-circulating pumps. The author describes different package holders and loading arrangements, and discusses liquor-flow characteristics in relation to the shape of the package. The uniformity of dyeing depends, among other factors, on the magnitude and uniformity of the winding tension (package hardness). Useful dyeing assistants include Lyogen DK (S) for direct and vat dyes on cotton. In the application of vat pigments, Stabiliser VP (Ciba) is useful. Albatex PO (Ciba) improves penetration and level dyeing. There are 27 illustrations. L.A.T.

Some Developments in Package Dyeing

W. H. Gambrell

Canadian Textile J., **78** (12 May 1961) 45–47

The following developments in package dyeing are discussed— (a) Dyeing of wool with reactive dyes at pH 4–5 in presence of a cationic condensate. (b) Production of two-tone effects on 100% wool by a one-bath method using the principle that reactive dyes in the absence of cationic agents will dye the tips and the abraded sections of the wool fibre while leaving the fibre body undyed, even after prolonged boiling. The body of the wool is dyed with milling acid dyes. (c) Levelling of milling acid dyes by using an agent of weakly basic character. (d) Dyeing with 1:2 metal complexes on Orlon by using large additions of salt. (e) Dyeing of nylon and high-bulk Orlon on sponges. (f) Dyeing of cotton with soluble sulphur dyes and with phthalocyanine blues. (g) Conversion of azoic dyeing recipes for different liquor ratios. W.P.M.

Modern Continuous Dyeing Processes

R. Kern

Amer. Dyestuff Rep., **50** (15 May 1961) P366–P373

Mechanisation and automation of textile processing in general is briefly reviewed. The use of continuous dyeing processes is discussed with particular reference to pad–steaming. A pressure steamer capable of working at 120°C (250°F) enables substantive dyes to be applied to cellulosic fabrics, and disperse, acid, and premetallised dyes to various synthetic-polymer fibres and blends. S.B.D.

Origin of Faults and Stains in Dyeing, Printing, and Finishing

P. Colomb *Teintex*, **26** (15 Apr 1961) 257–265

One of a series of articles. Discusses the origin of grease spots, tar spots, and weft stains; methods for their removal; and stain-repellent treatments for finished fabrics, with recipes. J.C.F.

Automatic Colour Control Applied to Hosiery Processing

J. S. Christie

Amer. Dyestuff Rep., **50** (17 Apr 1961) P287–P291

The 'Electrocolorset Process' for finishing nylon hosiery has been developed to scour, dye, finish, set, and dry hose in one continuous automatic operation. The development background is outlined for the machine, which is capable of processing 150 dozen/h, and the automatic colour-control system is discussed with emphasis on the conditions which must be held constant for reproducibility. S.B.D.

Homogeneous Acetylation of Cellulose dyed with Reactive Dyes

V. Einsele *Melliand Textilber.*, **42** (Apr 1961) 427–432

If cotton is dyed with reactive dyes, acetylated, and the acetic acid solution then poured into water, the dyed cellulose acetate is precipitated and very little dye goes into the aqueous phase. If the same experiment is repeated with cotton dyed with direct dyes, most of the dye leaves the precipitated cellulose acetate and goes into the water. When the rate of acetylation of cellulose dyed with reactive dyes is measured, it is found that dyes with one reactive group do not affect the rate but dyes containing two such groups (cross-linking effect) reduce the rate markedly. W.M.

Fixation of Dyes containing a Dichloro-s-Triazinyl Group on Cellulose

C. Preston and A. S. Fern

Chimia, **15** (Jan 1961) 177–186

The fixation on cellulose under alkaline conditions of a reactive dye of the type $Dye(Cl)_2$, can give rise to 4 types of products, viz., Cell·O·Dye·Cl, Cell·O·Dye·O·Cell, Cell·O·Dye·OH, Cell·O·Dye·NHR, derived from Cell-O·Dye·Cl by condensation with an amine NH_2R. The influence of fixation conditions on the proportions of these different types, and on the fastness of the resultant

dyeings to storage under humid, acid conditions, is described.   E.S.

Dyeing Mechanism of Reactive Dyes on Cellulose
H. Zollinger   *Chimia*, **15** (Jan 1961) 186–192
When fixing a reactive dye only a surprisingly small proportion is lost by combining with water present instead of with OH groups in the cellulose. Steric effects play an important part, the position of the dye molecule absorbed on the cellulose favouring reaction with the fibre.
E.S.

Dyeing with Vinyl Sulphone Reactive Dyes
H. Luttringhaus
Amer. Dyestuff Rep., **50** (3 Apr 1961) P248– P253
The basic reactions in dyeing with vinyl sulphone reactive dyes are—

(i) $Dye \cdot SO_2 \cdot CH_2 \cdot CH_2 \cdot OSO_3Na$ (unreactive, stable)
$\xrightarrow{alkali}$ $Dye \cdot SO_2 \cdot CH=CH_2$ (reactive)

(ii) $Dye \cdot SO_2 \cdot CH=CH_2 + HO \cdot Cell$ (fibre)
$\longrightarrow$ $Dye \cdot SO_2 \cdot CH_2 \cdot CH_2 \cdot O \cdot Cell$ (dyed fibre)

(iii) $Dye \cdot SO_2 \cdot CH=CH_2$ (reactive)
$\xrightarrow{water}$ $Dye \cdot SO_2 \cdot CH_2 \cdot CH_2 \cdot OH$ (unreactive)

Methods and results acceptable to the industry are discussed and shown. Based on practical experience, samples of actual mill runs are discussed and practical suggestions made for cotton and rayon packages; continuous equipment (e.g. curing ranges or pad–steam units); the jig: the Smith–Drum Cascade machine; and dyeing on reels. The practicability of dyeing from a long liquor ratio is discussed and the dyes suitable for this method are listed. The effect of resins on hue and light fastness is also discussed. While the dyes are especially designed for dyeing cellulosic fibres, practical results from mill runs are shown on man-made fibres (i.e. certain acrylic fibres; mixtures of acrylic fibres; blends of wool, acrylic fibres, and rayon; blends of high-tenacity rayon, nylon, and cotton), most of them by simple one-bath procedures.   S.B.D.

Reaction between Reactive Dyes and Cellulose
M. A. Chekalin   *Tekstil. prom.*, No. 1 (Jan 1961) 40–43
Filament viscose was dyed with a reactive dye of formula
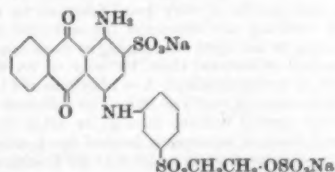
and subjected to a gradual hydrolysis using 58% sulphuric acid at room temperature. The main hydrolysis products were identical with those obtained by reaction of the dye with glucose and cellobiose. Further hydrolysis lead to separation of one and two glucose residues.   L.S.L.

The Procion–Resin Method
I. D. Rattee and R. Bugmann
S.V.F. Fachorgan, **16** (Mar 1961) 162–177
Paper presented at the General Meeting of the Swiss Association of Technical Dyers (S.V.F.) held in Zurich on 4 Feb 1961, giving a detailed account of combined application of Procion (ICI) dyes and resin for simultaneous dyeing and crease-resist treatment of cellulosic fabrics. In addition to the dye and resin pre-condensate, the padding liquor contains 10–25 g/l. Procion Resin Catalyst A, 3–50 g/l. Velan NW, and 3–5 g/l. Perminal PP. After drying (stenter) the resin is cured at 155°C for 3 min, and the fabric washed in two alkaline baths (3 g/l. soda ash and 1 g/l. Lissapol ND) at 95–100°C. The method is simple and economic and the results are reproducible,
L.A.T.

Selection of Direct Dyes for Application to Undesulphured Rayon Staple
A.A.T.C.C. Southeastern Section
Amer. Dyestuff Rep., **50** (17 Apr 1961) P299–P301
To study the relative dyeing characteristics of desulphured and undesulphured rayon staple, comparative

dyeings were made with 69 fast-to-light direct dyes, and evaluated for differences in colour and light- and wash-fastness. 43 Dyes gave equivalent shades and for all but three of these the light fastness of dyeings on both desulphured and undesulphured rayons was identical. The other 26 dyes gave significant undesirable colour changes on the undesulphured rayon and, in general, the dyeings on the fibre had lower light fastness than dyeings on desulphured rayon. The wash-fastness was not affected for any dye. All the 26 direct dyes which showed colour differences on undesulphured rayon were found to be metal complexes, and all the rest, except three, were not. The sulphur compounds *in situ* on the undesulphured fibres do not affect the colour of dyeings of the metal-complex dyes, but the sulphur or sulphur compounds must first become dispersed or dissolved in the dyebath before having an adverse effect on the colour.
S.B.D.

Dyeing Protein Fibres with Reactive Dyes—I
A. D. Virnik and M. A. Chekalin
Tekhnol. tekstil. prom., No. 6 (19) (1960) 109–115
Using paper chromatography, various chemical processes involved in the reaction of dichlorotriazine dyes with amino acids and with silk were investigated. Samples of commercial dyes were used; they were found to contain quantities of hydrolysed dye. During silk dyeing in mild alkaline medium at 50°C or mild acid medium at 85°C the whole of the dye which has not reacted with the fibre will be hydrolysed within 1 h. At pH 10·2 and 20°C traces of unhydrolysed dye are still present, even after 3 hours' dyeing. Under slightly acid conditions at 60°C no appreciable dye hydrolysis occurs after 4 h. In investigation of various functional groups of amino acids it was found that in mildly acidic conditions the dyes will react with amino and imino groups of α-N-acetylhistidine, but will not react with acylamino and hydroxy groups in either aromatic or aliphatic residues, the imino group of tryptophan or a guanidine residue. In mild alkaline conditions and at low temperature (20°C) the dyes will react with both aromatic and aliphatic hydroxy groups.
L.S.L.

Dyeing Unscoured Wool
L. I. Fidell
Amer. Dyestuff Rep., **50** (15 May 1961) 361–363;
(29 May 1961) 397–405
The normal scouring process is reviewed and the effect on the grease content compared with that of various steeping and spraying treatments and treatment with non-ionic detergent. Results of fastness tests are given and discussed for dyeings carried out on wool containing about 8% by weight of grease by the following sequence—spraying with water at 140°F (60°C), dyeing with 1:1 or 1:2 metal-complex, neutral-dyeing acid, or direct dyes, and scouring at 120–135°F (50–58°C) with a non-ionic detergent. Results of fastness tests for dyeings carried out on prescoured wool are also given.   S.B.D.

Dyeing Wool and Wool Mixtures at above 100°C
A. Würz   *Melliand Textilber.*, **42** (Apr 1961) 439–444
Temp. of 120–130°C are now widely used for the dyeing of polyester fibres. Wool–polyester fibre unions are popular and dyeing conditions to which wool can be exposed (temp., pH, time) without undue damage were studied. From the protective substances tested (degradation products of proteins, sulphite waste liquor, formaldehyde) formaldehyde (30%) is the most efficient, and addition of 5% is recommended. It appears that wool–polyester fibre unions may be dyed in presence of formaldehyde for up to 4 h at 105°C, 3 h at 110°C, 2 h at 115°C, and 1 h at 120°C. Loss of tensile strength is greatest at pH 7 and 2·8 and much less at pH 3·5 (resistance of wool is at a maximum near the isoelectric point).   W.M.

Effect of Temperature and pH on Adsorption of Basic Dyes by Wool
C. L. Bird and G. P. Stancey
J.S.D.C., **77** (June 1961) 244–246

Suspension Method in Wool Dyeing
V. F. Androsov
Tekhnol. tekstil. prom., No. 5 (18) (1960) 101–105
Wool fibre, fabric, and unions are treated with a suspension of insoluble acid dyes and the labile dyeing is fixed by heat and after-chroming, a wide range of colours

being obtained. The great advantage claimed is a 60–65% reduction in boiling time, resulting in less fibre damage. Dyeing recipes are given. G.J.K.

Evolution of Disperse Dyes and their Application to Hydrophobic Fibres
T. M. Baldwinson J.S.D.C., 77 (June 1961) 246–251

New Aspects of the Chemistry of Dyes: Dyeing of Synthetic Fibres
M. R. Gangueux Teintex, 26 (15 May 1961) 323–335
Review of the present position of dyes for polyamide, polyester, and polyacrylate fibres. J.C.F.

Recent Developments in Dyeing of Acrylic Fibres
B. Kramrisch J.S.D.C., 77 (June 1961) 237–244

Dyeing Nitron with Acid Dyes
P. V. Moryganov, B. N. Mel'nikov, and Z. N. Panina
Tekhnol. tekstil. prom., No. 1 (20) (1961) 106–112
Nitron (a polyacrylonitrile fibre) can be dyed with acid dyes after treatment with hydroxylamine, leading to the formation of carbamido groups. Max. dye absorption occurs during a relatively short period (5–10 min), prolonged dyeing resulting in decomp. of carbamido groups. Kinetic data on the absorption of Acid Red 2G, Red N, Rubine, and Fast Orange are tabulated. G.J.K.

Automatic Apparatus for Dyeing Nylon Stockings
G. Herbulot and H. Tusveld
Teintex, 26 (15 May 1961) 336–342
A brief description of the Teintofix 760 machine is given. The rotating carrier has four units, each of which carries 36 formers. The sequence of operations is as follows— (1) loading (and unloading), (2) pre-forming, (3) dyeing for 2 min at 95°C at a long liquor ratio, (4) drying. Laboratory investigations, under the conditions used in the machine, of the partition coefficient k of Nyloquinone dyes between fibre and dyebath, have shown that the dyes fall into two classes: those for which k increases with increasing dye concentration, and those for which k is independent of the concentration. A knowledge of k is required for maintenance of constant concentration in the dyebath, which is achieved by the addition of a concentrated dispersion after each batch. Two examples of actual machine operation are given. J.C.F.

Estimation of Affinity of Disperse Dyes for Polyesters
H. Hendrix Melliand Textilber., 42 (Apr 1961) 432–438
Disperse dyes were applied to polyester fibres using as dyebath mixtures of water and isopropanol in varying proportions. Maximum dye absorption occurs in most cases with 80% water, 20% alcohol. A certain amount of alcohol is necessary to dissolve the dye. At higher alcohol conc., bath and fibre compete for dye and absorption decreases. Diffusion experiments were carried out at temp. between 110 and 190°C by letting disperse dyes diffuse through a pile of polyester films and counting the number of layers actually penetrated. For anthraquinonoid dyes there is a correlation between the absorption maximum and diffusion, i.e. if the maximum is high many films are penetrated. W.M.

Swelling Action of Carriers in Dyeing Polyethylene Terephthalate Fibre
F. M. Rawicz, D. M. Cates, and H. A. Rutherford
Amer. Dyestuff Rep., 50 (1 May 1961) 320–323, 354
Water-insoluble carriers are shown to swell the fibre more than the soluble carriers and so are more effective on a weight basis. Swelling produces changes in the mechanical properties which would be expected for partial deorientation of the fibre; this was confirmed in one case by X-ray diffraction. Rate of dyeing of the treated and extracted fibre is greater than that of the untreated fibre, showing that swelling persists after removal of the carrier, although the effect is small compared with the total effect in presence of carrier. The action of an absorbed carrier is apparently to reduce the forces of attraction between polymer molecules. This allows greater mobility of polymer chains and consequent faster diffusion of the dye through the fibre. P.B.S.

Carrier Dyeing of Polyester Fibres
A. Shimonik Tekstil. prom., No. 1 (Jan 1961) 77–79

Dyeing Vinylon with Solacet Dyes. III— Dyeing with Binary Mixtures
A. Katayama, N. Kuroki, and K. Konishi
J. Soc. Textile Cellulose Ind. Japan,
17 (Apr 1961) 354–359
The absorption of binary mixtures of Solacet dyes by vinylon has been studied over a range of dye concentrations and temperatures (70, 80, and 90°C). Dyeings were carried out with 1:1 and 2:1 mixtures of Solacet Fast Yellow GS and Solacet Fast Blue 2BS, and 1:1 and 2:1 mixtures of Solacet Fast Blue 2BS and Solacet Fast Orange 2GKS. In dyeing with these mixtures, the amount of each dye absorbed is reduced by the presence of the other dye, but in all cases the total molecular concentration of the two dyes absorbed exceeds that of the individual dyes when applied alone. On the assumption that Solacet dyes, when applied as a mixture, are absorbed by vinylon without any mutual interaction, equations have been derived thermodynamically in order to correlate the amount of each dye absorbed with its affinity when applied alone. This affords a quantitative explanation of the reduction in the amount of dye absorbed, owing to the presence of the other component of the dye mixture. The affinity and heat of dyeing of each dye in the mixed dyeing have been determined, and these values are in reasonable agreement with those obtained when the dyes are applied alone. I.G.L.

Coloration of Cellulosic Textiles
ICI BP 866,160 (21 Nov 1956)
Colorations of extremely good fastness to washing are obtained by treatment with an alkali, a water-soluble heterocyclic compound containing two –N:C(Hal)– groups in the ring, and a dye containing at least one reactive amino group. Thus bleached cotton cloth is padded with its own weight of an aqueous solution containing 2% Solochrome Yellow 2GNS (C.I. Mordant Yellow 10), 2% 6 - (4′,6′ - dichloro - s - triazinylamino)naphthalene- 2 - sulphonic acid and 2% NaHCO₃. It is then steamed at 102°C for 5 min, rinsed and then soaped at the boil. This yields a yellow dyeing which is very fast to washing. C.O.C.

Anthraquinone and Azo Reactive Dyes for Cellulose
BASF BP 868,741 (Germany 19 Dec 1956)
Dyeings and prints of very good fastness to wet treatments and rubbing are obtained on cellulose by use of anthraquinone or azo dyes containing at least one –NR·AX group attached otherwise than by way of an SO₂ group (R = H, Alk, or halogenoalkyl; A = alkylene of 4 C and may contain OH groups; X = Cl or Br). The cellulose is treated with alkaline agents before, during, or after dyeing and printing and then, if necessary, heated for 1–10 min at > 100°C. Thus cotton cloth is padded at 30°C with a solution containing the yellow dye

$$HO_3S \quad \text{—N:N—} \quad N(CH_3)CH_2CH_2Cl$$

(15 g/l.) and NaCl (100), dried, padded with cold 1% NaOH, steamed for 3 min at 105°C, and soaped at the boil. The resulting dyeing has very good fastness to washing and rubbing. C.O.C.

Dyeing Cellulose Textiles
Ciba BP 868,492 (Switzerland 14 Aug 1956)
The material is padded with an organic dye containing at least one acid, water-solubilising group and at least one aliphatic halogenated acylamino group and then heated or steamed in presence of an acid-binding agent. Thus cotton cloth is padded with 75% of its own weight of a solution at 80°C containing water (100), urea (9), and the dye

$$ClCH_2CH_2\cdot CO\cdot NH— \quad SO_3H \quad NH_2 \\ \text{—N:N—} \\ HO \quad SO_2H$$
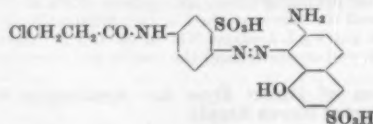
(1), dried, impregnated with 75% of its weight of an aqueous solution containing 1% NaOH and 30% NaCl, steamed for 60 s at 100–101°C, rinsed, treated with

NaHCO$_3$, and boiled for 15 min in a 0·3% aq. soln. of a non-ionic detergent. This yields a red dyeing fast to boiling.   C.O.C.

Dyeing Cellulose with Reactive Dyes

Ciba   BP 867,521 (Switzerland 10 Aug 1956)

The materials are treated at 20–50°C with an aqueous solution of a chromiferous or cobaltiferous azo dye containing at least one 2,4-dichloro-1,3,5-triazine radical, and the dye is then fixed by treatment with an acid-binding agent whose aqueous solution is more alkaline than that of Na$_2$CO$_3$, e.g. NaOH.   C.O.C.

Fast Dyeings on Cellulosic Textiles with Dyes containing a Reactive Amino Group

Ciba   BP 869,549 (Switzerland 4 Sept 1957)

Use of a dye containing at least one water-solubilising group and at least one reactive amino group, accompanied by treatment during or after dyeing with a compound containing two epoxy groups or with 2,3-dibromobutane-1,4-diol and then heating, results in dyeings of good fastness. Thus cotton cloth was impregnated with an aqueous solution containing the dye 1-aminobenzene-2-sulphonic acid $\xrightarrow{\text{alkali}}$ 2-amino-5-hydroxynaphthalene-7-sulphonic acid (0·43 g/l.), NaOH (0·2), and butadiene dioxide (1·8), dried at 80°C and heated for 10 min at 140°C, rinsed, and soaped at the boil; the dyeing remained unaffected. When the process was carried out using no butadiene dioxide the dye was completely removed from the fibre during soaping.   C.O.C.

Pad Dyeing of Cellulosic Textiles

Ciba   BP 867,546 (Switzerland 14 Aug 1956)

Good dyeings are obtained by use of a dye containing at least one water-solubilising acid group incapable of splitting off easily and at least one substituent which splits off easily with the taking over of the electron pair of the bond and which is linked to a C atom of an alkyl sulphone group, the dye being fixed by steaming in presence of an acid-binding agent. Thus cotton is impregnated with an aqueous solution of the dye

OCH$_3$

–N:N–C–CH$_3$

HO–C N

SO$_2$

CH$_2$CH$_2$Cl

N

SO$_3$H
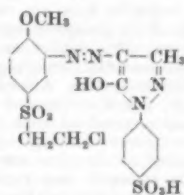
dried, then impregnated with aq. 30% brine containing 1% NaOH, steamed for 60 s at 100–101°C, rinsed in water, rinsed in aqueous NaHCO$_3$ and boiled with a non-ionic detergent for 15 min. This yields a strong reddish yellow dyeing of excellent fastness to washing.   C.O.C.

Level Dyeing of Wool with Reactive Dyes containing a Chloro-s-triazinylamine Group

ICI   BP 869,150 (8 Oct 1958)

Use of an aqueous bath containing the dye, an acid or acid salt, a cationic surfactant, a non-ionic dispersing agent and an amine, alcohol, or phenol gives good exhaustion and level dyeing.   C.O.C.

Dyeing Wool with 1:2 (Cr or Co) Complexes of Monoazo Dyes

Ciba   BP 869,056 (Switzerland 14 Nov 1958)

Dyeing is carried out in presence of a wool-resist agent having affinity for the fibre, e.g. tannin or HCHO–naphthalene sulphonic acid condensates, and hexamethylenetetramine. One dye molecule of the complex used must be free from sulphonic and carboxylic groups not participating in the complex formation. The dyebath must be at pH < 7, at least at the start of dyeing.

         C.O.C.

Simultaneously Dyeing all Fibres in a Wool-Cellulosic Fibre Fabric

Ciba   BP 867,168 (Switzerland 14 Nov 1958)

The dyebath contains acid and direct dyes, a wool-immunising agent and hexamethylenetetramine and must have pH < 7, at least at the start of dyeing. Thus a fabric having a spun viscose rayon warp and a wool weft is introduced at 50°C into an aqueous bath containing crystalline Na$_2$SO$_4$ (40), 40% acetic acid (6), and a mixture of a naphthalene sulphonic acid–HCHO condensate (1) and hexamethylenetetramine (2). It is worked at that temperature for 15 min and then a 1:2 metal-complex acid dye and a direct dye are added, the bath is brought to the boil in 30 min, and dyeing continued at the boil for 1 h. Shading can be carried out with either dye without affecting the levelness of the dyeing.   C.O.C.

Dyeing Cellulose Triacetate

Celanese Corpn. of America

         BP 867,607 (U.S.A. 25 Sept 1956)

The dye is applied from solution in a bath composed of water, a monohydric aliphatic alcohol, an alkali-metal or NH$_4$ thiocyanate and, if desired, acetic acid. The amount of thiocyanate should be such as to cause fibres of acetone-soluble cellulose acetate of acetyl value 54–55% to coalesce but not to damage cellulose acetate fibres of acetyl value < 60%. A wide range of dyes is applicable by this method, e.g. disperse, solubilised vat, direct, acid, and 1:2 metal-complex dyes.   C.O.C.

Azoic Dyeing of Cellulose Triacetate

BrC   BP 867,202 (27 Nov 1956)

Applying an azoic diazo component, diazotising, and coupling with 2,3-hydroxynaphthoic acid gives much quicker coupling if the fibres are water-swollen during the coupling. The fibres are brought into this state preferably by combined action of water and a softening agent, e.g. diethylene glycol diacetate.

         BP 867,203 (28 Nov 1956)

Navy blues to blacks are obtained by applying 4,4′-diamino-2-acetylamino-5-methoxyazobenzene, diazotising, and coupling with 2,3-hydroxynaphthoic acid.   C.O.C.

Dyeing or Printing Polyethylene Terephthalate Fibres

Ciba   BP 867,535 (Switzerland 8 Feb 1957)

Dyes of formula

Y$_{n-1}$

X$_{m-1}$ –N:N–C–C–R^2

NO$_2$ HO–C N

N

R^1
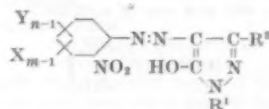
(X and Y = Hal, Alk, cyanoethyl, or alkoxy of < 5 C, or trifluoromethyl; n and m = 1, 2, or 3; R^1 = H, Alk, or cyanoalkyl of < 7 C, or C$_6$H$_5$ which may be substituted by Cl atoms and/or CH$_3$ groups if R^2 = H, Alk of < 5 C or carboxyalkyl (Alk of < 5 C)) have good affinity for polyester fibres from aqueous dispersion. They are especially suitable for use in conjunction with a carrier or for high-temperature dyeing.   C.O.C.

Fast Black Dyeings on Linear Polyesters

FH   BP 870,045 (Germany 15 Jan 1957)

Very fast black dyeings on linear polyester fibres are obtained by absorbing into the fibre a polynuclear, especially a binuclear, black base suitable for oxidation dyeing (e.g. 4-aminodiphenylamine, 2,4′-diaminodiphenylamine, and mixtures thereof with, e.g. benzidine and especially α-naphthylamine, aminophenols, and diaminobenzenes) and developing with acidified hypohalite solution or preferably with acidified aq. soln. or dispersion of a compound hydrolysing to hypohalous acid, or with an acidified chlorite solution. Thus, scoured Terylene (4·39) is treated for 15 min at 85°C in an aq. bath (50:1 liquor ratio) containing 15% 4-aminodiphenylamine (I) (on wt. fibre), 3 g/l. o-phenylphenol, 1·5 g/l. anion-active alkylphenyl polyglycol ether sulphate, and a little glue as stabiliser. Dilute NaOH is added to bring to pH 8·5 and after boiling for 2 h the dyeing is rinsed and developed for 15 min at 60°C in 4 g/l. sodium toluene-4-sulphonic acid-N-chloramide and enough acetic acid to give pH 4·0. A deep black dyeing of good fastness to light and washing is obtained. I may also be applied at higher temperatures in absence of carrier, e.g. 3 h at 140°C.   A.T.P.

Applying Azoic Dyes to Polyester Fibres at above 100°C

FH   BP 869,631 (Germany 20 Nov 1956)

Azoic dye components are applied to polyethylene terephthalate fibres in presence of a solution promoter

(e.g. tetrahydronaphthalene) and a substance which on heating gradually lowers the pH (e.g. ammonium salts or organic esters), as described in *BP* 809,221 (J.S.D.C., **75** (1959) 270), but using temperatures above 100°C (e.g. 120°C). Treatment in hot (80–85°C) aq. nitrous acid then develops the azoic dye.   E.S.

Dyeing Polyolefins
British Nylon Spinners   *BP* 868,869 (22 Jan 1959)
Those basic dyes suitable for dyeing acrylic fibres are also useful for dyeing polypropylene fibres.   C.O.C.

Producing Designs on Fabrics by Embossing and then Dyeing
United Merchants and Manufacturers
BP 868,458 (U.S.A. 9 Sept 1958)
The fabric is led through the nip formed by an embossing bowl and a smooth bowl, i.e. one which has not been "run in". This results on subsequent dyeing in a two-tone colour effect being obtained.   C.O.C.

Fixing Pigments on Fibrous Materials
FH   *BP* 866,767 (Germany 21 Aug 1956)
Modification of *BP* 810,548 (J.S.D.C., **75** (1959) 331). Improved fastness to washing and dry cleaning is obtained by using a composition containing (*a*) a pigment, (*b*) an alkaline solution of a polycarboxylic acid–polyhydric aliphatic alcohol condensate containing reactive carboxyl groups, (*c*) a compound containing $>$ 1 reactive ethylene-imine radical or its homologue or derivative, and (*d*) a water-soluble precondensate obtained from melamine or a phenol or urea. Fixation is obtained by steaming or dry heat at $>$ 70°C, preferably $>$ 100°C.   C.O.C.

Fixing Pigments on Foils and Fibrous Materials
FH   *BP* 868,935 (Germany 6 July 1956)
The foils or fibrous materials are treated either simultaneously or one after the other with a pigment and an aqueous solution or dispersion of a carboxylic compound of mol. wt. $>$ 800 and one of formula

$$R \cdot OP : \left(N <^{CH_2}_{CH_{2'}} \alpha \right)_2$$
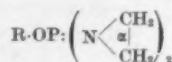
(α = homologues or derivatives; R = primary aliphatic amine of 4–10 C, primary cycloaliphatic amine, aliphatic, aromatic or cycloaliphatic hydrocarbon, –NXY (X = Alk or cycloalkyl; Y = Ar or cycloalkyl) or

$$-N <^{X}_{Y})$$

(X = Alk; Y = Ar or cycloalkyl; X is linked to Y to close a heterocyclic ring) and then steamed or heated at $>$ 100°C. The resultant dyeings or prints are very fast to washing.   C.O.C.

Developments in Textile Auxiliaries (III p. 450)

Interaction between Acid Wool Dyes and Nonyl Phenol–Ethylene Oxide Derivatives (IV p. 452)

Relation between Constitution, Colour and Light-fastness of Basic Dyes of the Indazole Series on Acrylic Fibres (IV p. 452)

Printing and Dyeing Pile Fabric (IX p. 467)

IX— PRINTING

Investigation of Properties of Thickeners in Printing Pastes— III
Sin Men Khen and F. I. Sadov
Tekhnol. tekstil. prom., No. 6 (19) (1960) 102–108
The stability of structure of various thickeners has been determined. The process of gradual destruction of this structure in the colour box, which begins before pressure has apparently been exerted on the paste, has been followed. The effective changes in structure for various thickeners and their mixtures have been determined for various printing times. Thixotropic properties of paste and their dependence on various mechanical factors, e.g. speed of the printing roller, have been also investigated.   L.S.L.

Transfer of Dyes on to Fabric during Machine Printing
Sin Men Khen and F. I. Sadov
Tekstil. prom., No. 1 (Jan 1961) 36–39
The transfer of dye to the fabric is a very complicated process depending on various chemical and physical properties of printing pastes. The printing pastes used in experiments were based on Rongalite, alkali, Vat Bright Green C and a number of thickeners. Rheological, structuro-mechanical, and thixotropic properties and the adhesive power of each paste were determined. Freshly prepared paste, because of its undamaged and firm structure, has a high and stable viscosity. The same paste in a colour box will be characterised by a certain amount of damage to its mechanical structure, by its thixotropic properties and its adhesion to metal. During actual printing the behaviour of paste is determined by low viscosity, as a result of mechanical damage of molecular structure, pressure between printing roller and furnishing roller, adhesive power between paste and fabric, and finally paste ductility. When on fabric, the paste is characterised by its thixotropic properties.   L.S.L.

Rationalisation in Roller Printing
A. Franken   *Chemiefasern*, **11** (May 1961) 338–341
Various machines suitable for laboratory printing control are reviewed, the advantages of such control enumerated and some technical details given. Laboratory printing machines (e.g. the four-colour Model ML 44 of SACM) enabling shades to be checked, new thickeners, etc. tried out, a test printing machine for checking on the rollers before use, i.e. new rollers or those stored for some time (e.g. Test Printing Machine System G. W. Mettenheimer), and laboratory steamers are discussed. Means of preventing damage to the engraving rollers during storage, transport, etc. are also considered.   I.G.L.

Printing Wool with Cibacron Dyes
W. Badertscher
S.V.F. Fachorgan, **16** (Mar 1961) 194–198; (May 1961) 351–352
Cibacron (Ciba) reactive dyes are capable of forming covalent links with –NH$_2$ groups of wool substrate. The colour yield is approx. 50% higher on chlorinated wool than on the untreated material. Dye migration is inhibited by the use of suitable thickeners. After fixing in saturated steam for 10–15 min, prints are rinsed in water, then in dilute ammonia, and neutralised with formic acid. The May issue contains figures showing— (*a*) dependence of tensile strength of wool on urea concentration in bath and (*b*) effect on tensile strength and yield of variation in sodium bicarbonate concn. when working with wool unions.   L.A.T.

Advances in Printing of Polyester–Cotton and Polyester–Wool Fabrics
G. Bertolina and A. Broggi
Melliand Textilber., **42** (Apr 1961) 449–451
Mixtures of disperse and reactive dyes are recommended for use on polyester fibre–cotton fabrics. For dye fixation, high-temperature treatment (Thermosol process) is applied. To obtain good penetration, mixtures of emulsion and alginate thickeners are most suitable. To avoid staining of white grounds, addition of products with affinity for dyes (Albigen A) to the first rinsing bath is advisable. Ratio of disperse to reactive dye should be between 2 : 1 and 3 : 1 to obtain best fastness properties. For dye fixation, treatment at 195–200°C for 45–60 s is required. Lists of suitable disperse and reactive dyes are given. In printing of polyester–wool fabrics, leuco-esters of vat dyes have good affinity for wool, but only moderate affinity for polyester fibres. To obtain uniform results addition of small amounts of disperse dyes is recommended. Steaming is done at 17–18 lb/in^2 pressure for 90 min. For re-oxidising, peroxide and sulphuric acid are used. Lists of suitable Indigosol and disperse dyes are given.   W.M.

Fixing Vat and Sulphur Dye Prints
British Cotton Industry Research Assocn.
BP 865,659 (26 July 1957)
Modification of *BP* 848,792 (J.S.D.C., **77** (Feb 1961) 84). Marking-off is prevented and the method made applicable

for use on standard machines by sandwiching the printed material between two travelling bands of impervious material as it passes against the cylinders.　　C.O.C.

Printing and Dyeing Pile Fabric
C.S.C.M.　　　　　　　　BP 865,415 (U.S.A. 13 Aug 1957)

The fabric with its pile downwards is pressed downwardly against a flat receptacle having a thin erect wall with free edges defining the design. This places an area of the pile in the receptacle and separates it from the remainder. The receptacle is supplied with a measured quantity of colorant and the pile within the receptacle is compressed to make it absorb all the colorant. A number of receptacles may be used to form the design, each receptacle having its own supply of colorant, which may be the same as or different from that supplied to other receptacles.

　　　　　　　　　　　BP 865,416 (U.S.A. 13 Aug 1957)

The fabric passes under a pressure roller to press it against the receptacles which are formed on a carrier surface moving in a closed path.　　C.O.C.

Spirit Duplicating yielding Copies of Excellent Fastness to Light
Columbia Ribbon & Carbon Manufacturing Co.
　　　　　　　　　　　BP 867,507 (U.S.A. 19 July 1956)

The image on the master sheet is formed by an organic colour-donor compound containing $\not<$ 1 benzene ring and $\not<$ 2 OH groups occupying adjacent positions in one of the rings, e.g. Logwood Extract (C.I. Natural Black 1). The copy sheets contain a metallic compound which forms an intensely coloured compound with the colour-donor compound, salts of Fe, Va, and Cu being especially effective. The carrier in which the colour-donor compound is applied to the master sheet is such that when the sheet is wetted with the solvent it allows a small portion of the colour-donor compound to be dissolved and transferred to a copy sheet placed against it.　　C.O.C.

Hectograph Carbon Paper using Azoic Coupling and Diazo Components
General Aniline　　　　　　USP 2,950,213 (31 Aug 1956)

A base sheet is coated with a wax composition containing the complex of an azoic coupling component with a liquid, strongly basic amine, e.g. the complex of C.I. Azoic Coupling Component 2 with dicyclohexylamine were, together with diazotised 4-benzoyl-2,5-diethoxyaniline, stabilised with methyl taurine incorporated into a wax hectograph composition and coated on to paper. Hectograph master sheets were then prepared in the usual manner and further copies obtained on copy paper from the master sheet using as solvent a 9:1 mixture of alcohol and glacial acetic acid. The blue dye developed readily on the copy paper and copies could be used immediately without set-off or smearing.　　C.O.C.

Diazotype Materials Sensitised with N-Hetero-p-aminobenzenediazonium Salts
General Aniline　　　　　　USP 2,948,613 (26 July 1957)

Much improved image density, opacity, and brilliance are obtained by using as sensitiser a benzenediazonium salt containing, para to the diazo group, a 1,3-oxazolidino or 1,3-oxazino group, e.g. the diazonium salt from p-oxazolidinoaniline hydrochloride.　　C.O.C.

Two-component Diazotype Materials yielding Prints with no Background Discoloration and Free from Sulphur Stabilisers
General Aniline　　　　　　BP 867,432 (U.S.A. 22 Sept 1958)

Use of a diazo component containing a phenyl group substituted by a heterocyclic nitrogenous ring system containing a hetero-O atom, e.g. p-morpholinobenzenediazonium chloride, and as coupler a derivative of phenol or resorcinol, e.g. m-hydroxyphenylurea, yields diazotype intermediate prints whose background does not discolour on ageing, despite there being no sulphur stabiliser present.　　C.O.C.

Multicontrast Photographic Emulsions containing Cyanine Dyes
Eastman Kodak Co.　　　　　USP 2,947,631 (18 Mar 1957)

Photographic silver halide emulsions showing varying degrees of contrast are obtained by incorporating in them an oxonol dye containing a 3-methyl-1-sulphophenyl-2-pyrazolin-5-one or a barbituric acid nucleus and as sensitiser either a cyanine dye containing a 5-phenyl-benzothiazole nucleus or a carboxyalkyl sulphoalkyl group, or a merocyanine dye containing a carboxyl or sulpho group, or 4 - [1,3,3 - trimethyl - 2(3H) - indolylidene)-ethylidene]-3-methyl-1-phenyl-2-pyrazolin-5-one.
　　　　　　　　　　　　　　　　　　　　　　　C.O.C.

Positive Diazotype Copies
Chemische Fabriek L. van der Grinten
　　　　　　　　　　　BP 867,629 (Holland 5 Feb 1957)

Diazotype material containing a compound of formula

$$XN{:}N\!-\!\langle\!\!\!\bigcirc\!\!\!\rangle\!-\!NR^1R^2$$
$$OR^3$$
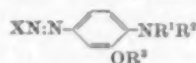
(X = anion; R^1 = Alk of < 5 C; R^2 = Alk of < 3 C or aralkyl of < 8 C; R^3 = Alk of < 5 C) is exposed to a mercury vapour lamp. The visually observable end-point of the exposure lies particularly near to the point at which the diazotype material has been exposed just sufficiently in practice. In addition the exposure time for a given image density is much shorter than with hitherto known combinations of diazo compounds and light sources.

　　　　　　　　　　　BP 867,630 (Holland 4 June 1958)

Diazo compounds of formula

$$Y$$
$$XN{:}N\!-\!\langle\!\!\!\bigcirc\!\!\!\rangle\!-\!NR^4R^5$$
$$OR^6$$
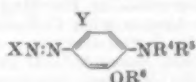
(R^4 and R^5 = same or different Alk, hydroxyalkyl or chloroalkyl of < 5 C or aralkyl and nuclearly substituted chloroalkyl groups of < 12 C; R^6 = Alk of < 5 C or aralkyl of < 12 C; $R^4 + R^5 + R^6$ contain 6–17 C). They are more active couplers than those described in the preceding abstract, yield darker azo dyes, and in one-component diazotype material cause less bleeding during semi-wet development.　　C.O.C.

Subtractive Colour Film
E. Gretener　　　　　　BP 867,517 (Switzerland 16 July 1956)

A method of producing a subtractive colour film in which the black–white density required for dosing the brightness is recorded in a black printer and the colour residues necessary for colour reproduction are recorded in three-colour registrations.　　C.O.C.

Four-colour Photographic Prints
Edgar Gretener　　　　BP 867,518 (Switzerland 15 May 1957)

Describes production of prints comprising a neutral grey-black component and three colour-residue components, the colour components at least being of the variable area type.　　C.O.C.

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X— SIZING AND FINISHING

Interaction of Coating Material and Base Fabric Structures
F. K. Burn　　Canadian Textile J., 78 (14 Apr 1961) 58–63

The factors influencing pile bonding, yarn shifting, handle, and durability are discussed, and methods of controlling the coating process are described.　W.G.C.

Effect of Chemical Treatment on Weathering of Spun Dress Fabrics
M. M. Dianich
　　　　　Tekhnol. tekstil. prom., No. 5 (18) (1960) 96–100

Samples of spun tricot mélange dress fabric were prepared in 20% dimethylolurea (I), dimethylolurea plus hydroxymethylstearylamide (II), and 10% hydrophobic silicon organic emulsion (III) by padding and heat

treating. The tearing strength, elongation, and work of rupture for each were determined before and after treatment and after 3 months' summer weathering in L'vov. The tabulated results show the superiority of III in tearing strength over I and II. The loss in work of rupture of all treated fabrics is less than the loss of the untreated fabric after weathering.   G.J.K.

Study of Hypochlorite-resistant Melamine-type Finishes

S. L. Vail, J. G. Frick, and J. D. Reid

Amer. Dyestuff Rep. **50** (15 May 1961) 356–360

Commercial finishing agents based on melamine–formaldehyde products are widely used but many exhibit chlorine retention, causing yellowing of fabrics. Yellowing has been attributed to the reaction of hypochlorite with primary amino groups in the finish, since the placing of stable substituents on each amino nitrogen of the s-triazine ring before reaction with formaldehyde eliminated yellowing. The properties of fabrics treated with the formaldehyde reaction product of acetoguanamine, *N,N*-bis(2-hydroxyethyl)melamine, *N,N'*-dimethyl - *N'',N'''* - bis(2 - hydroxyethyl)melamine, and *N,N',N''*-trimethylmelamine are presented and compared with those for fabrics treated with some commercial methylolmelamines and methylated methylolmelamines.
S.B.D.

Inter- and Intra-molecular Bonding in Reaction of Crease-recovery Agents with Cellulosic Fabrics

S. J. O'Brien and W. J. van Loo

Text. Research J., **31** (Apr 1961) 340–348

The relation (R) between improvement in crease recovery and the concentration of a number of crease-recovery agents applied under different conditions to cellulosic fabrics of different structures and composition is similar to the relation in the formaldehyde–cotton system. That only a small fraction of the amount of agent required to impart a given improvement in crease recovery is involved in cross-linking is true for all types of cross-linking agents. The relation between the improvement in crease recovery and the concentration of agent needed to give wet crease recovery only is similar to that between improvement in dry crease recovery and concentration of agent that has reacted. The structure and weave of a cotton cloth have little influence on R. Cyanoethylation of cotton makes the cellulose hydroxyl groups less accessible, but R is of the same form as for unmodified cotton. Cross-linking the wet and swollen fibre to impart wet but not dry crease recovery does not prevent subsequent cross-linking to obtain improved dry crease recovery, but the tendency to form cross-links is reduced. When the physical character of cellulose differs from that in cotton, as it does in rayon, the difference in R is more profound. Here the constants in the theoretical R differ in a way which indicates that the tendency of the agent to cross-link is less in rayon than in cotton.   C.O.C.

The Catalyst in the Cellulose–DMEU Reaction. I— Effect of Catalyst on Physical and Chemical Properties of Finished Cottons

H. M. Ziifle, R. J. Berni, and R. R. Benerito

Text. Research J., **31** (Apr 1961) 349–365

Probably the catalyst forms a complex with DMEU through formation of nitrogen-to-metal bonds. The final pH depends not on the catalyst but rather upon the formaldehyde evolved. Crease-recovery properties and chlorine retention depend upon the catalyst concentration and there is a critical catalyst concentration at which maximum cross-linking, maximum crease recovery, and minimum chlorine retention are obtained. At this concentration MgCl₂ and Zn(NO₃)₂ gave the same degree of protection against chlorine damage, Mg(NO₃)₂ was less effective but better than ZnCl₂. No correlation was found between the analytically determined chlorine retention and observed damage, nor was there detectable loss of the total chlorine retained as a result of chlorination followed by scorching. Consistent and reproducible differences in infrared absorption spectra caused by DMEU treatment do not distinguish between catalysts but do distinguish between treatment in absence or in presence of a catalyst. Laundering does not affect the infrared absorption spectra, does not remove the metallic ion, and causes no loss in nitrogen content when a catalyst is used. Absorption spectra of chlorinated and of chlorinated and scorched

specimens in the 3–15 μ region did not reveal presence of N–Cl bonds. Apparently damage during the chlorine scorch test is caused by a free-radical mechanism in which the metallic complex is an important factor. Changes in the absorptivities of DMEU-treated specimens seem to indicate that the treated fabric is oxidised during laundering and that the nature of the oxidation depends upon the catalyst used. These changes suggest that cross-linking occurs by reaction of the DMEU with the primary alcohol group of the cellulose.   C.O.C.

Catalysis of the Reaction of Urea–Formaldehyde Precondensates on Cellulose

R. Steele

J. Applied Polymer Sci., **4** (July–Aug 1960) 45–54

The reaction of dimethylolurea with cotton cellulose is catalysed by ammonium chloride and by sodium carbonate. Cross-linking takes place with ammonium chloride as a catalyst, leading to changes in resilience and other physical properties and increased resistance to acid hydrolysis. Cross-linking does not take place if sodium carbonate is used or if monomethylol urea is used.   P.T.S.

Formaldehyde Treatment of partly swollen Cotton

L. H. Chance, R. M. Perkins, and W. A. Reeves

Text. Research J., **31** (Apr 1961) 366–376

Study of the reaction of HCHO with cotton at various degrees of swelling has led to development of two cotton fabrics having different physical properties. The first fabric was produced by the batch-wise Form-W process in which the wet, swollen fabric is treated in an aqueous solution of HCHO and HCl. The resulting fabric has excellent wet crease recovery and is suitable for line-drying after washing. Its physical properties are similar to those of most commercial wash-and-wear cotton fabrics. The treated fabric is stable to repeated laundering, acid souring, and chlorine bleaching. The second fabric was produced by the batch-wise Form-D process. Here the wet, partly swollen fabric is treated in an aqueous solution of acetic acid, HCl, and HCHO. The treated fabric has good wet and dry crease recovery and can be either line- or tumbler-dried. It has a soft handle and is stable to repeated laundering, acid sour, and chlorine bleaching. In both processes it is important that the fabrics be first well mercerised if they are not to lose unduly in strength. In Form-D process only 5–10 min are needed at 45°C to obtain the same crease recovery properties as are obtained in 1 h at 27°C. In both processes only methylene bridges are formed.   C.O.C.

Changes in Physical Properties of Cotton on Cross-linking

F. H. Steiger, S. Y. Wang, and M. D. Hurwitz

Text. Research J., **31** (Apr 1961) 327–339

Cotton reacted with formaldehyde by dry curing shows a poor crease recovery–strength relationship which is primarily a function of the formal linkage and not of the degradative effects of acid. Inconsistencies in results obtained with formaldehyde are associated with the slow rate of hemiformal formation in the padding liquor. Wet cotton can be cross-linked with hemiacetal linkages by treatment with glyoxal. The loss in strength caused by cured dimethylolethylene urea at a given level of crease recovery is comparable to that obtained from uncured glyoxal in absence of the degradation associated with the hot acidic curing of thermosetting finishes. The ratio of wet to dry crease recovery suggests that the areas of cellulose and/or the hydroxyl groups involved when cotton is cross-linked in glyoxal solutions are the same as those involved in the dry curing of cross-linking agents. High wet to dry crease recovery ratio requires that cross-linking at some stage should take place when the fibre is swollen more than it is in the application of typical aqueous crease-recovery agents for cotton. That different areas and/or hydroxyl groups are involved in the attainment of wet and dry crease recovery is supported by the fact that dry crease recovery can be imparted to cotton independently of a previously imparted high wet crease recovery.
C.O.C.

Cross-linking Reactions in Cellulosic Fibres. I— Organic Chemistry, Heterogeneity, and Accessibility

D. D. Gagliardi and F. B. Shippee

Text. Research J., **31** (Apr 1961) 316–327

All known cross-linking agents contain groups which can form ethers or esters with cellulose hydroxyl groups.

Because of the physical limitations imposed by the state of the cellulose during processing and the various side reactions which can occur with any compound, it is apparent that all known methods of dimensionally stabilising and imparting recovery from creasing to cellulosic fabrics involve heterogeneous modification of the cellulose fibres. General statements cannot be made about the extent of cross-linking produced by all compounds and the role played by the side reactions. With all compounds, if cross-linking is prevented from occurring, then little or no modification of fibre properties affecting dimensional stability and crease recovery is produced. As long as cross-linking occurs the side reactions do not prevent production of dimensional stability and crease recovery. If, however, there is present a hydroxylated material in addition to the cellulose, the cross-linking agent can react with either or both. As the concentration of the second material increases, the gain in weight first increases and then decreases and at very high concentrations the fabric remains the same as if it had never been treated. In such cases the fibres have acted simply as the reaction vessel in which the cross-linking agent has reacted preferentially with the second hydroxylated material. Only a small proportion of the total hydroxyl groups in cellulose are involved in present cross-linking treatments. Thus at low ratios of cross-linking agent to cellulose, cross-linking is favoured. As the amount of the agent increases, the cellulose hydroxyl groups (available to that compound) become saturated and no further reaction with the fibre occurs. After such saturation the excess of agent may remain unreacted in and on the fibres and be removed in subsequent scouring. If, however, the compound contains reactive groups then reaction with itself can begin to take place to give resins or other products.   C.O.C.

Properties of Cotton Fabrics treated with Fluoro-carbon Combinations with Water-repellent Agents

H. B. Goldstein   *Text. Research J.*, **31** (Apr 1961) 377–387

The beneficial effects of the Quarpel finish may be obtained at significantly lower cost by using about 5% of fluorochemical and 16% of quaternary water-repellent agent. Use of the mixture greatly improves the water-repellency and resistance to oil-borne stains. In addition, it imparts a fairly high level of rot-resistance. The finish is faster to washing, dry cleaning, and abrasion than that obtained with either of the agents separately. Appropriate choice of combination enables white fabrics to be treated with little or no yellowing. The whiteness and soil-resistance obtained by use of the mixture are unaffected by short periods of weathering, although there is a slight drop in the spray rating.   C.O.C.

Contribution of Swelling to Water-resistant Cotton Fabrics: Effects of Maturity of Cotton, Cloth Construction, and Finishing Processes.

C. G. Goldthwait and W. G. Sloan

Text. Research J., **31** (May 1961) 434–446

Fabrics which become water-impermeable because their constituent fibres swell when they are wetted tend to lose some of their swelling capacity during finishing, this being more marked with fabrics made from immature cotton than with those made from mature cottons. Entrapped air in partly swollen fabrics contributes to water resistance beyond the effect of air in ordinary water-repellent goods. This is especially true of cloths swollen in water vapour and may add to the resistance of a fabric in service if exposed to a very humid atmosphere.   C.O.C.

Treatment of Wool with Chlorosulphonated Poly-ethylene

R. E. Whitfield   *Text. Research J.*, **31** (May 1961) 446–451

Polymers of chlorosulphonated ethylene can be grafted on to wool by curing at 130–150°C for 6–24 h. Under milder conditions of cure only 1–2% resin is grafted. Although deposition of ≮ 8% resin on wool without grafting gives good resistance to shrinkage and washing, grafting is necessary to make the finish fast to dry-cleaning. In addition, grafting of the polymer yields the desired properties at lower resin contents. The treated fabric is little altered in handle, crease-recovery, abrasion, tear strength or colour, but shows increased tendency to soil.   C.O.C.

Introduction of Vinyl Groups into Wool

L. A. Miller and R. E. Whitfield

Text. Research J., **31** (May 1961) 451–455

Wool was vinylated by treating it with acrylyl chloride, maleic anhydride, *N*-carbamyl maleimide, ethyl acrylate and glycidyl methacrylate. The degree of vinylation and the siting of the vinyl groups in the fibre can be controlled by varying times, temperatures, and solvent and reagent concentration. With fibre-swelling solvents, e.g. water, acetic acid, and dimethylformamide, reaction probably occurs throughout the entire fibre, but with non-swelling solvents reaction seems to be limited to the fibre surface. The vinylated wool looks the same as the untreated wool with the exception that wool treated with acrylyl chloride is slightly yellowed and that treated with *N*-carbamyl maleimide becomes slightly pink.   C.O.C.

Heat- and Steam-setting of Wool Yarns

B. Mihalik and I. Asbóth

Textil Praxis, **16** (Feb 1961) 167–172

After reviewing the basic chemistry of temporary and permanent set, the authors describe the steam-setting of wool yarns in hank and package form. Hanks were steamed at 100, 110, 120, and 130°C for 10, 20, 30, and 60 min in saturated steam. The yarn was dried at room temp. and allowed to condition before further testing. It was found that breaking strength and elongation diminished as the temp. and duration of steaming increased. Above 120°C the effect of time of steaming is far more critical than at lower temp. Chemical modification of the wool fibre was assessed by the acid and alkali solubility tests. Both parameters increased with higher temp. and longer times of treatment. Yellowing of wool was also temp. dependent— 60 min steaming at 100°C produced similar effect to 10 min at 130°C. The effectiveness of steaming was checked by twist liveliness tests. Usually satisfactory results can be obtained by steaming at 110°C for 30 min, and these conditions should not be appreciably exceeded to avoid tendering of wool. In order to assess the uniformity of steaming, cops of yarn were treated at 130°C for 15, 30, and 60 min. The solubility and dyeing tests showed little difference between the inner and outer layers at shorter times of steaming. As the duration of steaming increased these differences became more marked.   L.A.T.

Competition as the Driving Force in Synthetic-fibre Finishing— II

J. G. Evans   *Chemiefasern*, **11** (May 1961) 342–343

Following Part I (*J.S.D.C.*, **77** (Aug 1961) 394), which was concerned with improvements in creasing properties, developments and trends in the finishing of cellulose acetate fabrics and in methods of imparting good water- and dirt-repellency, inflammability, and stability to heat to cellulose acetate and viscose rayon are reviewed.

I.G.L.

PATENTS

Animalisation of Cellulose

American Cyanamid Co.   USP 2,947,594 (9 Dec 1957)

Cellulose is given affinity for wool dyes by treating it in presence of an alkaline catalyst with a monomeric quaternary ammonium compound having a terminal vinyl or $CH_2{:}CH$ group, e.g. 2-vinyl-*N*-methylpyridinium hydroxide.   C.O.C.

Delustring

BrC   BP 865,857 (1 Nov 1956)

Textiles, especially cellulose acetate, are impregnated with an aqueous solution of a melamine–formaldehyde condensate, a non-ionic dispersing agent and, as catalyst, an acid of dissociation constant ≮ 1 × 10⁻⁴ or a salt thereof with a volatile base, and then heated so that they are not dried. This gives uniform and reproducible delustring. Thus an acetate crêpe fabric was impregnated with an aqueous solution containing methylolmelamine, citric acid, and Dispersol VL (ICI), passed through a steam chamber at 80°C for 4–9 min and scoured. The treated fabric was delustred, the effect being fast to washing and dry cleaning. It had a pleasant, soft but "weighty" handle.   C.O.C.

Acetylating Rayon so that it retains its Fibrous Structure
Société de la Viscose Suisse
BP 868,019 (Holland 2 May 1958)

The rayon is swollen by water and the water removed from the fibres by treating them with glacial acetic acid. They are then activated by treatment with glacial acetic acid containing acetic anhydride and $>$ 0·2% perchloric acid on the dry weight of cellulose. Excess of activating liquid is removed and the fibres are acetylated for $\geqslant$ 6 h at 10–25°C with a liquid made up of CCl_4 (1500–3000% on dry wt. of cellulose), acetic anhydride (300–500), and perchloric acid (0·1–0·5).   C.O.C.

Imparting Crease-recovery Properties to Cellulosic Fibres
O. Jackson   *BP* 866,387 (19 July 1958)

Enhanced crease-recovery is obtained by giving two separate impregnations with the materials which impart crease-recovery properties, the material applied by the first impregnation being cured before the second impregnation is made. At least one of the impregnations is made with HCHO and a catalyst.   C.O.C.

Rendering Woven Cellulose Textiles of Low Cover Factor Resistant to Corrugation in Washing and Tumbler Drying
Chicopee Manufacturing Corpn.
USP 2,945,738 (29 Jan 1958)

Acetals of b.p. $>$ 125°C prepared by the process of *USP* 2,785,947 are dissolved in water, padded into woven cellulose textiles of low cover factor, dried, and baked. This results in the textiles becoming resistant to corrugation during washing and tumbler drying without detracting from their softness and absorbency.
                    C.O.C.

Preventing Pilling in Fabrics of Wool–Synthetic-polymer Fibre Blends
Société Rhodiaceta   *BP* 868,166 (France 28 Nov 1958)

Fabrics containing $\leqslant$ 35% by wt. of synthetic-polymer fibres in the blend are brushed, cropped, and milled in that order. If desired they may be singed between cropping and milling. This yields flannel-type fabrics which do not pill.   C.O.C.

Raising the Safe-ironing Temperature of Cellulose Triacetate
BrC   *BP* 869,605 (7 Dec 1956)

Treatment with isopropyl alcohol, esters of monohydric aliphatic alcohols, e.g. methyl acetate, acetic and propionic acids, caprolactam, γ-butyrolactone, urea, C_1 to C_4 acid amides, aqueous acetone containing a thiocyanate with or without an alcohol, and solutions of benzoic or other benzene carboxylic acid in C_1–C_4 aliphatic alcohols, is given at $<$ 100°C until the safe-ironing temperature is raised by $\leqslant$ 10 deg. C.   C.O.C.

Stabilising Polyester Fibres to Ultraviolet Radiation
General Aniline   *BP* 867,262 (U.S.A. 26 May 1958)

When the fibres are treated at 340–350°F for 5–15 s with a diethylene glycol solution of a compound of formula

$$R^1O\!-\!\langle\rangle\!-\!\underset{O}{\overset{OH\ HO}{\underset{\|}{C}}}\!-\!\langle\rangle\!-\!OR^2$$
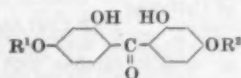
(R^1 = H or Alk of 1–5 C; R^2 = Alk of 1–5 C), e.g. 2,2′-dihydroxy-4,4′-dimethoxybenzophenone, and rinsed with water, the ultraviolet absorber is completely dissolved in the fibres so that it is fast to washing. On subsequent dyeing the protection is also given to the dye, so that dyes may be used that hitherto affected the rate of degradation of the fibres or were themselves insufficiently fast to light.
                    C.O.C.

Stabilising Tubular-knit Fabric
Compax   *BP* 868,207 (U.S.A. 8 Jan 1958)

The fabric is set to width by spreading it out flat and steaming it while it is flat. It is then immediately fed into a compressive zone which it leaves at a lower speed than that at which it enters.   C.O.C.

Compressive Shrinking
Bradford Dyers' Association   *BP* 866,074 (23 Nov 1956)

The shrinking apparatus and the Palmer dryer are run at such speeds that the speed at which the fabric leaves the shrinking apparatus is only slightly less than that at which it passes around the dryer. The fabric passes straight from the shrinking apparatus to the dryer without coming into contact with any roller or other supporting surface. The length of this unsupported run is so short that in travelling through it the fabric is stretched at most by a percentage not greater than the percentage difference between the speed at which it leaves the shrinking apparatus and that at which it travels around the dryer. This enables a given degree of shrinkage to be more uniformly and conveniently obtained than hitherto and, in addition, in a shrinking apparatus with a rubber sleeve, wear and tear on the sleeve is reduced to a minimum.
                    C.O.C.

Shrinking or Stretching Flexible Sheet Material
S. F. James   *BP* 865,599 (30 June 1959)

The material to be treated is placed on several narrow members whose upper surfaces are roughened or serrated so that they engage the underside of the material when it is pressed on to them. These members are so supported and interconnected that they can be all moved so that they can have narrow gaps between or can be brought into contact with one another so as to eliminate the gaps. The material is pressed on to the members by any suitable means and the members are moved towards or away from one another so as to shrink or stretch the material as desired.   C.O.C.

Applying Metal Films to Textiles and other Sheet Material
Heberlein & Co.   *BP* 869,076 (Austria 14 May 1957)

Decorative patterns of non-porous glossy metallic film are obtained by applying an adhesive in the necessary pattern to the cloth, applying a transparent foil having a vacuum-coated metal layer on the side applied to the cloth, and then drawing off the transparent foil. This gives a much greater gloss than is obtained by direct vacuum-coating of the cloth.   C.O.C.

Antistatic Finish
Boehme Fettchemie   *BP* 868,809 (Germany 29 Aug 1957)

An antistatic finish of good fastness to washing is obtained by use of a hardened water-soluble or water-dispersible condensate of an amino triazine or one of its derivatives, a keto compound, and a polyalkylene oxide, e.g. the condensate of melamine (1 mol.), 30% HCHO (6) and polyethylene oxide of mol. wt. 1000 (2).   C.O.C.

Controlling Tension during the Heat Stretching of Nylon Tyre Cord
Firestone Tire & Rubber Co.
USP 2,947,060 (21 Sept 1955)

The fabric passes through one or more groups of rolls, each group consisting of pairs of rolls with each pair driven at a speed different from that of the preceding pair so that change in tension is distributed throughout each group of rolls. This minimises roll wear and obviates damage to the fabric during stretching.   C.O.C.

Coating Polyester and Nylon Fabrics with Polyurethanes
ICI of Australia and New Zealand
BP 868,500 (Australia 2 Apr 1957)

The composition used consists of an organic isocyanate (5–30% by wt.), a partially condensed polyester (50–70), a vinyl terpolymer (5–40), a plasticising polyester (0·2), and a catalyst (0–5). Its use on lightweight fabrics imparts improved flexibility and resistance to teasing and ageing. Heavyweight coated fabrics obtained by its use have longer service life.   C.O.C.

Finishing the Edges of Coated Fabric
Firestone Tire & Rubber Co.
BP 866,945 (U.S.A. 14 Aug 1957)

Bonded-fibre Fabrics
Interchemical Corpn.   *USP* 2,949,386 (3 Nov 1958)

Bonded-fibre fabrics of good fastness to washing and having a good handle are obtained by using as binder a latex of a polymer containing carboxyl groups and a cross-linking agent for the polymer.   C.O.C.

Continuous Lamination
Armoride   *BP* 867,776 (France 30 Aug 1956)

A moving web is impregnated with a resin, heated to at least partly cure the resin, and a second web is

applied to the rear face of the first web. The webs are then passed through a bath of resin during which they are separated. They are then brought together and passed in close contact against a large-diameter heated drum with the first web nearer the drum surface. They are compressed firmly together on the drum and drawn off as a laminate.
C.O.C.

Lamination
Polymer Corpn. *BP* 867,792 (U.S.A. 4 June 1957)
At least one of the materials, preferably both, to be laminated is led through a fluidised bed of a solid adhesive, the materials during passage through the bed being at a temperature at least as high as the sintering temperature of the adhesive particles and preferably above their m.p. This causes some of the adhesive to adhere to the material. The components are then pressed together.
C.O.C.

Photochemical Degradation of Nylon 6.6 Treated with Size Based on Acrylic Acid (VI p. 461)
Origin of Faults and Stains in Dyeing, Printing, and Finishing (VIII p. 462)
Automatic Colour Control Applied to Hosiery Processing (VIII p. 462)
Effects of Softeners on Thermoplastic Resins (XIII p. 472)
Photochemical Modification of High Polymers by Visible Light (XIII p. 473)

XI— PAPER AND OTHER CELLULOSIC PRODUCTS

New Methods of Modifying Cellulose
Z. A. Rogovin *J. Polymer Sci.*, 48 (Dec 1960) 443–456
The preparation of cellulose phenyl ethers, esters with amino acids, chloroalkanoic acids, and phosphorus acids are described. Cellulose can be prepared with aldehyde, carboxyl, amide, nitrile, and amino groups. It is possible to make graft copolymers with cellulose and acrylonitrile, a polyamide, or a polyether by choosing the appropriate cellulose derivative and monomer. P.T.S.

Interaction of Cellulose with Glycerol and Ethylene Carbonate
E. Wellisch, L. Hagan, L. Marker, and O. J. Sweeting
J. Applied Polymer Sci., 3 (May–June 1960) 331–337
The equilibrium moisture content of cellulose films softened with hygroscopic glycerol and with non-hygroscopic ethylene carbonate was determined as a function of the softener content at various relative humidities. P.T.S.

Catalysts for Acetylation of Cellulose
C. J. Malm, L. J. Tanghe, and J. T. Smitt
Ind. Eng. Chem., 53 (May 1961) 363–367
The efficiency of perchloric, sulphuric, methane-sulphonic, methanedisulphonic, and sulphoacetic acids, when used as catalysts in acetylation, has been studied from reaction rate measurements using a thermal technique. Systems in which these catalysts were used included acetic anhydride and water (I) or methanol (II), hydroxyls of a cellulose ester soln. (III), cotton linters (IV), and regenerated cellulose (V). In case of (I) no difference in reaction rates was found between perchloric and sulphuric acid catalysts, whereas system (II) required three times the amount of sulphuric acid needed in I owing to its conversion to methyl hydrogen sulphate. (III) required a greater spread of the relative amounts of catalysts (compared with perchloric acid) in order to give equal reaction rates. Acetylation of (IV) was most efficient when sulphuric acid was used, because of retention of this acid at the sites of acetylation, whereas catalysis of system (V) was less efficient since cellulose acetate hydrogen sulphate, which was first formed at the surface of the cellulose, passed rapidly into soln. F.J.

Retention of Crystalline–Amorphous Ratio of Cellulose during Heterogeneous Acid Hydrolysis
J. J. McKeown and W. I. Lyness
J. Polymer Sci., 47 (Nov 1960) 9–17
Heterogeneous hydrolysis of cellulose is supposed to attack amorphous regions rapidly and then the ordered regions of crystallites much more slowly. Thus hydrolysis should slowly increase the amount of crystalline material

in samples by destroying the non-crystalline material. X-ray diffraction studies of the products of hydrolysis of cellulose from several sources show that this is, in fact, not the case. P.T.S.

Grafting on to Cellulosic Macromolecules through Chain Transfer to Mercaptoethyl Side Chains. I— Experimental Procedure and Results
D. K. Ray Chaudhuri and J. J. Hermans
J. Polymer Sci., 48 (Dec 1960) 159–166
Cellulose acetate films, Cellophane, and cellulose fibres were mercaptoethylated by addition of ethylene sulphide. The effects of reaction time, catalyst concentration, and monomer concentration (styrene or methylmethacrylate) on the rate of formation of the grafted polymer were observed. P.T.S.

Cross-linking Cellulose Derivatives by High-energy Radiation
F. C. Leavitt *J. Polymer Sci.*, 45 (Aug 1960) 536–538
Below a critical viscosity, solutions of cellulose derivatives have sufficient freedom of molecular movement for the cross-linking reaction under the influence of high-energy radiation to compete with degradation reactions. A network structure is formed and gelation takes place. P.T.S.

Grafting on Cellulose Acetate
G. Faraone, G. Parasacco, and C. Cogrossi
J. Applied Polymer Sci., 5 (Jan–Feb 1961) 16–22
Describes introduction of an unsaturated group into the cellulose ester and grafting vinyl polymers on to this group. It is possible to graft 6–8% polystyrene by this method. P.T.S.

Oxidation of Dihydroxycellulose with Nitrogen Oxides
N. Ya. Lenshina, V. S. Ivanova, and V. I. Ivanov
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, No. 3 (Mar 1961) 519–521
The oxidation of dihydroxycellulose with nitrogen oxides has been followed by determining the content of uronic and non-uronic carboxyl groups. The total carboxyl-group content increases to a maximum of about 26% during the first 24 h and then remains constant. The dihydroxy group is decomposed with the formation of glycolaldehyde, which is rapidly oxidised to glycollic acid, the presence of which has been identified chromatographically. A reaction mechanism is suggested. I.G.

Mixtures of Casein and Synthetic Latex for Coating Paper
G. Genin *Lait*, 40 (1960) 627–633
Chem. Abs., 55 (15 May 1961) 9872
Casein and soyabean protein were used with Dow latex in the manufacture of coated and pigmented papers. The pick resistance of the pigmented coatings was greatest when the latex was the sole adhesive. Replacing part or all of the latex by casein or soyabean protein lowered the pick resistance. Mixtures of the latex and the protein produced coatings of higher brightness. As the proportion of adhesive increased, printability with ink decreased, and the decrease was greater as the amount of protein in the adhesive increased. C.O.C.

Hydrocarbon Resins in Paper Coatings
P. O. Powers and J. F. Pflum
Ind. Eng. Chem., 53 (May 1961) 371–374
The uses and properties of polyterpene resins, prepared by polymerisation of β-pinene, and polydiene resins polymerised from aliphatic dienes and olefins are described. Both are low-mol.-wt. polymers and are largely hydrogenated cyclic structures with no aromaticity, and are similar in properties. They may be used as hot-melt, solvent soln., emulsified, or powdered resin coatings. As additives to polyethylene and polypropylene they improve coating characteristics by giving reduced viscosity, lower moisture transmission rates, better adhesion, and improved pigment dispersion. F.J.

PATENTS

Stable Neutral or Acid Aqueous Solutions or Dispersions of Alkali-soluble Cellulose Ethers
American Viscose Corpn. *USP* 2,947,645 (29 Oct 1957)
Cellulose ethers soluble in 0·5–4·0% aqueous NaOH remain in solution when the NaOH is completely neutralised or the neutral solution is brought to pH 5–9.
C.O.C.

Paper containing Titania (C.I. Pigment White 6)
Johnson & Johnson   *USP* 2,947,658 (10 July 1956)
Adding a water-soluble solution of reactive titania to a dilute paper stock containing an impregnant latex results in the paper formed from the stock having improved wet tensile strength, delamination resistance and tear strength and adequate elongation and flexibility. The water-soluble reactive titania solution can be prepared from a water-soluble orthotitanic ester in presence of a suitable screener to delay hydrolysis of the orthotitanate thus producing a highly reactive form of monomeric or low-polymeric orthotitanic acid.   C.O.C.

Acrylamide Polymers for rendering Paper Resistant to Penetration by Grease, Oil, and Water
Dow Chemical Co.   *USP* 2,945,775 (18 Apr 1955)
Application of water-soluble high-mol.-wt. polymers of acrylamide to paper imparts increased receptivity. Increased gloss ink hold-out and grease resistance result with a simultaneous increase in resistance to feathering and penetration by water-based inks.   C.O.C.

Wet-strength Paper by Use of a Linear Polyamide Resin
American Cyanamid Co.   *USP* 2,948,652 (9 June 1958)
Paper of improved wet and dry strength is obtained by adding to the pulp 0·5–1·5% (on dry wt. of fibres) of a water-soluble cationic thermosetting methylene–bisacrylamide – alkylenepolyamine – cyanate – formaldehyde resin and drying the paper at 190–250°F.   C.O.C.

Paper having Good Wet Strength
Monsanto Chemical Co.   *USP* 2,949,396 (23 May 1956)
Water-dispersible, acid-aged, cationic, sulphite-modified melamine–formaldehyde resins which, before acid ageing, contain 2·5–4·5 mol. of HCHO and $>$ 0·2 mol. of sulphonate groups per mol. of melamine and have viscosity $\not<$ 300 cp in 55% aq. soln. at 25°C added to the fibre slurry impart high wet strength to the resulting paper without any need for ageing or high-temperature treatment.   C.O.C.

Mineral-filled Paper
S. D. Warren Co.   *USP* 2,949,397 (13 June 1955)
Coating the filler particles completely or partly with a substituted mannan (a complex polysaccharide, see Vol. 7 of *Encyclopedia of Chemical Technology*, Interscience, N.Y. 1951) renders use of alum to prevent loss of filler unnecessary.   C.O.C.

High-gloss, Cast-coated Paper
Mead Corpn.   *USP* 2,950,214 (25 Mar 1957)
An aqueous, pigmented coating composition containing a pigment binder is applied to the paper which is then passed through a coagulating bath so as to obtain complete gelation of the binder. The coating is then passed over a press roll and transferred to a casting surface, e.g. a methyl silicone resin casting surface chemically bonded to a metal substrate. This results in a paper having unusually high brightness, opacity and gloss, and enhanced affinity for printing inks.   C.O.C.

Sequin-impregnated Paper
Paper Supply Co.   *USP* 2,949,398 (22 Nov 1955)
Sequins of metal or plastic foil are incorporated into the fibre slurry while it is in an agitated state before being discharged on to the cylinder or wire screen of the paper making machine.   C.O.C.

High-gloss Coated Paper
Consolidated Water Power & Paper Co.
          USP 2,949,382 (28 Feb 1958)
High-gloss paper of improved brightness, opacity, smoothness and enhanced printability is obtained by giving a fast coating of a mineral pigment composition having high adhesive content, so as to cover the fibres, fill the interstices and impart strength. A second coating of mineral-pigment composition having a low adhesive content is then given. This second coating remains on the surface of the paper where it is levelled to provide a remarkably smooth surface.   C.O.C.

Reaction between Urea and Cellulose (VI p. 459)

Shrinking or Stretching Flexible Sheet Material (X p. 470)

Applying Metal Films to Textiles and other Sheet Material (X p. 470)

Photochemical Modification of High Polymers by Visible Light (XIII p. 473)

Instrumentation— Its Success and Delusions (XV p. 476)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Continuous Production of Sheeting containing Leather Fibres and an Elastomeric Binding Agent
Michigan Research Laboratories and Electro-Chem Fiber Seal Corpn.   *USP* 2,948,692 (1 May 1957)
An aqueous suspension of shredded leather is treated with an ethylene oxide–dicyandiamide condensate and then with a curable elastomeric latex. It is then formed into a sheet, e.g. on a papermaking machine, and cured.
          C.O.C.

Structural-mechanical Properties of Protein Fibres (VI p. 460)

Photochemical Modification of High Polymers by Visible Light (XIII p. 473)

XIII— RUBBER; RESINS; PLASTICS
Some Difficulties in Coloration of Plastics
J. M. J. Estevez   *J.S.D.C.*, **77** (July 1961) 300–303

Effects of Softeners on Thermoplastic Resins
F. Magnussen
          Canadian Textile J., **78** (12 May 1961) 37–40
The alteration in physical properties occurring on 150-den. secondary acetate lining fabric when finished with four different thermoplastic resins in combination with seven types of softeners, was investigated. The properties investigated were— sewing properties, stiffness, abrasion, and tear strength. Very different physical properties are obtained when different softeners are used with thermoplastic resins, and it is possible to upgrade fabric performance by correct choice of resin and amount of softener while still retaining a satisfactory handle.
          W.P.M.

Degradation of Rubber by Chemical Agents in Solution
G. H. Foxley   *Proc. I.R.I.*, **8** (Apr 1961) 63–72
The action of various classes of compounds as peptisers for rubber solutions is reviewed and the mechanisms involved are considered. Degradation of rubber in solution may proceed via several mechanisms all of which involve free radicals. Although oxygen assists degradation its presence is not always essential.   W.R.M.

The Xenon Lamp: An Evaluation of the Tendency of Rubber Compounds to Discolour on Exposure to Light and of PVC Colours to Fade
A. G. Buswell   *Trans. I.R.I.*, **37** (Apr 1961) 43–51
Methods of assessing the tendency of a white rubber compound to discolour on exposure to light are considered. Differences resulting from exposure in the open, under glass, and under Perspex are considered. The discoloration obtained on exposure out of doors is compared with those obtained on exposure to a xenon lamp, mercury vapour lamp, and carbon arc source. Discolorations obtained with the xenon lamp correlate fairly well with those obtained on out-of-doors exposure. There is reasonably good correlation between fading of pigments incorporated in PVC when exposed out of doors and when exposed to a xenon source.   W.R.M.

Preparation and Properties of Aqueous Dispersions of Vulcanised Synthetic and Natural Rubber
B. A. Dogadkin, G. N. Zachesova, and I. A. Shokhin
          Kolloid. Zhur., **23**, No. 2 (Mar–Apr 1961) 150–156
The preparation of fine aqueous dispersions of synthetic and natural vulcanised rubber by the incorporation of an oleophilic emulsifier and its subsequent saponification with aqueous alkali is described. The degree of dispersion depends upon the clearance between mastifying rollers, their temperature, and the kind of emulsifier used. The use of a plasticising agent (Renacite 2, containing 42·5%

of trichlorothiophenol), which reacts with free radicals formed during the mechanical and thermal treatment of rubber, lowers the average particle diameter in dispersions.

I.G.

Electron-microscopic Investigation of Formation of Colloidal Particles of White Fillers in Latices

A. P. Narinskaya, A. P. Pisarenko and, B. V. Shtarkh

Kolloid. Zhur., 23 (2) (Mar–Apr 1961) 177–179

Fine dispersions of fillers, e.g. carbonates and aluminates, in latices can be obtained only in the presence of surface-active agents, in particular when the filler is precipitated in latex medium. The tenacity of vulcanised rubber containing highly disperse fillers is superior to that of rubber with large aggregates of fillers.　　I.G.

Backing, Coating, and Bonding with Rubber Latex

P. G. Cook　　*Canadian Textile J.*, 78 (14 Apr 1961) 53–57

The compounding and use of natural and synthetic rubber latices in the backing of carpets, fabrics, etc. are described.　　W.G.C.

Photochemical Modification of High Polymers by Visible Light

G. K. Oster and G. Oster

Mezhdunarod. Simpozium po Makromol. Khim., Doklady, Moscow, (1960) Sektsiya 3, 289–292 (*in English*)

Chem. Abs., 55 (15 May 1961) 9012

Polymers containing labile H, e.g. polyvinyl alcohol and mercapto polymers, can undergo H abstraction and cross-linking in visible light, while polyethylene requires far ultraviolet. Dyes, e.g. thiazine, fluorescein and 3,6-diaminoacridine dyes, are used as they undergo photo-reduction in presence of electron donors, e.g. allylthio-urea, ascorbic acid, cysteine, or chelating agents, e.g. ethylenediaminetetra-acetic acid and triethanolamine. Thus a thiolated gelatin can serve as an electron donor to the dye when the SH groups in the gelatin are oxidised in air with formation of SS cross-links. The cross-links can be destroyed by cleavage with ultraviolet radiation (254 mμ). Another type of cross-linking arises from the complexing of a metal ion with two neighbouring polymer molecules. This type is reversible and involves merely change in the valence state of the metal ion. The photo-reduced dye can reduce the ions Fe^{3+}, Ti^{4+}, Cr^{2+}, CrO_4^{2-}, Ag^+, MoO_4^{2-} and Hg^{2+} in presence of chelating agents which serve as electron donors to the light-excited dyes but do not reduce the metal ions. Chemical processes whose reactivity depends on a particular valence state of these metal ions can thus be controlled by visible light, e.g. salts of Cr^{3+} cross-link many polymers, including polyvinyl alcohol, polyacrylamide, all proteins, and many polysaccharides. These salts can be formed by dye-sensitised photoreduction of the dichromate ion. Thus cross-linking of these polymers can be controlled by visible light. This explains and extends the application of the long-known dichromate process of photoengraving. Similar results can be obtained with Hg, which is a cross-linking agent in its higher rather than in its lower valency state.　　C.O.C.

Sorption of Binary Solvents by Butadiene–Acrylonitrile Copolymers

G. L. Starobinets and V. F. Tikavyi

Kolloid. Zhur., 23 (1) (Jan–Feb 1961) 112–117

The sorption of binary systems of the type solvent–polar non-solvent (benzene/aliphatic alcohols) and solvent–non-polar non-solvent (aromatic hydrocarbons/aliphatic hydrocarbons) by butadiene–acrylonitrile vulcanisates containing 18, 26, and 40% acrylonitrile has been investigated. The distribution of binary system components between polymer phase and liquid phase, and the sorption potentials of the components, have been determined. The sorption potential of the solvent in systems containing polar non-solvents decreases with increasing content of nitrile groups in the copolymer. In the case of systems containing non-polar non-solvent the opposite is true.

I.G.

Kinetics of Caprolactam Hydrolysis in Aqueous Solutions of KOH

M. I. Vinnik, Yu. V. Moiseev, and L. V. Palagina

Doklady Akad. Nauk S.S.S.R., 138 (1) (May–June 1961) 149–152

The catalytic activity of aqueous solutions of alkalis at 25–90°C has been investigated by studying spectro-photometrically the kinetics of caprolactam hydrolysis in aqueous solutions of KOH. Caprolactam concentrations from 3×10^{-3} to 6×10^{-3} mol/l. and KOH concn. of 1–46% were used. Rate of hydrolysis is not proportional to concentration of OH^-. The catalytic activity of KOH solutions may be represented by the equation $K_{eff}/a_{H_3O^+} a_\pm =$ const., where $a_\pm$ is the average ionic activity of KOH solutions.　　I.G.

Hydrolysis of Polymethacrylates

A. D. Yakovlev and Z. S. Sokolova

Zhur. priklad. khim., 34 (Feb 1961) 464–466

The resistance of poly-n-alkyl methacrylates to alkaline hydrolysis increases with an increase in (a) chain-length of the alkyl group and (b) mol.wt. of the polymer. Methyl, ethyl, butyl, and octyl deriv. have been investigated.

G.J.K.

Stereoregular Polymers. VII— Infrared Spectra of Isotactic Polystyrene. Isotactic Poly-α,β,β-tri-deuterostyrene, and Isotactic Poly-p-deuterostyrene

H. Tadokoro, Y. Nishiyama, S. Nozakura, and S. Murahashi　　*Bull. Chem. Soc. Japan,* 34 (Mar 1961) 381–391 (*in English*)

The considerable changes of infrared spectra which the title cpd. afford with crystallisation, and their infrared dichroism, are now reported in detail. Methylene group vibrations and phenyl group in-plane and out-of-plane vibrations are discussed at length.　　H.H.H.

Normal Co-ordinate Treatment and Assignment of Infrared Absorption Bands of Polyvinyl Chloride

T. Shimanouchi and M. Tasumi　　*Bull. Chem. Soc. Japan,* 34 (Mar 1961) 359–365 (*in English*)

Since recent developments in high-speed computers have enabled the calculation of the normal vibrations of complex mol., the frequencies of the syndiotactic polyvinyl chloride mol. are now determined using the force constants from simple mol. The results suggested modifications of the assignment proposed in former studies (cf. Krimm and Liang, *J. Polymer Sci.*, 22 (1956) 95; Narita, Ichinohe, and Enomoto, *ibid.*, 37 (1959) 273, 281) and these are now reported together with the method of calculation.

H.H.H.

Flame-retardant Agent for use with Polymers

Dow Chemical Co.　　*BP* 867,468 (U.S.A. 24 July 1958)

Polybromocyclohexanes containing > 2 Br and < 4 Cl atoms, the total Hal being 6, e.g. tribromotrichlorocyclo-hexane, when incorporated into polymers render them flame-retardant and/or self-extinguishing. They also impart stability against discoloration on heating or exposure to light.　　C.O.C.

Colouring Plastics with Metal Pigments

Eastman Kodak Co.　　*USP* 2,947,646 (7 Jan 1958)

A thin coating of metal is deposited on to the finely powdered plastic which is then worked into the plastic state with or without additional plastic or other ingredients e.g. plasticisers. This results in all the metal particles being < 0·1 μ in diameter. The appearance obtained varies from pale transparent colours to opaque, the colour and its intensity depending on the metal, its average particle size, degree of dispersion, concentration and the thickness of the plastic sample being viewed.　　C.O.C.

White Sheets of Polymeric α-Chloroacrylic Esters

General Aniline　　*USP* 2,947,036 (17 Jan 1957)

Treating polymers of an α-chloroacrylic ester with steam at 250–260°F and above atmospheric pressure yields a finely foamed product which is an opaque white but yet translucent so that it resembles white porcelain or marble.

C.O.C.

Coating Rubber

Dunlop Rubber Co.　　*BP* 867,509 (17 Apr 1957)

If a linear polymer having isocyanate end groups is applied as a thin layer to rubber it sets to form an adherent coating on exposure to the atmosphere or heat. Presence of a cross-linking agent is unnecessary.　　C.O.C.

Polyester Filaments and Fibres having a Metallic Lustre (VI p. 461)
Applying Metal Films to Textiles and other Sheet Material (X p. 470)
Hydrocarbon Resins in Paper Coatings (XI p. 471)
Sulphonation and Sulphation (XV p. 476)

XIV— ANALYSIS; TESTING; APPARATUS

Photomicrographic Microscope for Both Micro and Macro Application
G. F. Norris, F. H. Meiller, K. T. Bolen, and L. R. Crisp
J. Opt. Soc. Amer., 51 (Apr 1961) 445–446
A simple instrument for preparations up to 4×4 in. with a magnification of $2600 \times$. E.COATES

Automatic Photometer for Measuring Angular Dissymmetry of Light Scattering
F. J. Baum and F. W. Billmeyer Jr.
J. Opt. Soc. Amer., 51 (Apr 1961) 452–456
A block diagram of the optical system and a detailed description of the component parts are given, of an automatic photometer for the recording of the dissymmetry of light scattered in the angular range 20–160°. The instrument is primarily designed for the measurement of the molecular weight and associated parameters of high polymers. The instrument employs a unique optical system and a cylindrical scattering cell of new design. E.COATES

Determination of Double Bonds by Reversed-phase Paper Chromatography
D. McHale, J. Green, and S. Marcinkiewicz
Chem. and Ind., No. 17 (29 Apr 1961) 555–556
A practical confirmation of theoretical postulates that compounds having the same structural differences will give constant differences in R_M when chromatographed under identical, near-ideal conditions, e.g. ΔR_M is constant for any monoene and its saturated counterpart. In practice, if any unsaturated compound is catalytically hydrogenated and samples are removed and chromatographed from time to time, each reduction product will yield a separate band, the number of bands being equal to the number of double bonds. Thus, Vitamin A benzoate (R_f 0·82), hydrogenated and chromatographed using liquid paraffin as stationary phase and 95% ethanol as mobile phase, yielded 5 bands having R_f values 0·73, 0·64, 0·56, 0·46, and 0·35. A.J.

Some Aspects of the Relation between Molecular Structure and Ultraviolet Spectra
B. Pullmann *Chimia*, 15 (Jan 1961) 4–9
Whereas in a purely hydrocarbon conjugated system, λ_{max} is due to a $\pi \rightarrow \pi^*$ transition, in a system containing hetero atoms, it is generally due to an $n \rightarrow \pi^*$ transition. A wide range of polycyclic and heterocyclic structures is discussed. Thus, hydrogenation, especially in a conjugated system, causes a bathochromic displacement of λ_{max} whereas phenylation of non-benzenoid aromatic hydrocarbons gives, in absence of steric effects, a hypsochromic displacement. A.T.P.

Optical Properties of Coloured Colloidal Systems. II— Apparent Refractive Index and Extinction Coefficient of System of Small Spherical Particles
M. Nakagaki and T. Fujii *Bull. Chem. Soc. Japan*, 34 (Mar 1961) 433–436 (*in English*)
The theoretical values of apparent relative refractive index m' of coloured particles are calc. as a function of particle size for various values of m_0 and k_0, where $m' = \mu_2/\mu_1 = m_0 - ik_0$, and k_0 shows the absorption of light. The values of m' and also of x (bulk) $= (4/3) k_0 = a$ quantity proportional to the extinction coeff., for infinitesimally small particles, show that m' increases to a limiting value with increases of k_0, while x approaches zero after passing a max. The values of m' and x are given for soln. of Acid Orange R (C.I. 15575) from measurements with a modified Brice-type differential refractometer and by a spectrophotometer. A method to convert m' and x into m_0 and k_0 values is illustrated (cf. *Ibid.*, 31 (1958) 980). H.H.H.

Volumetric Estimation of Dimethylphenylbenzyl-ammonium Chloride (Leucotrope O)
Z. I. Blagoveshchenskaya
Tekhnol. tekstil. prom., No. 6 (19) (1960) 116–119
A method based on precipitation of Leucotrope O with potassium ferrocyanide in sulphuric acid has been developed. L.S.L.

CIE System of Colour Measurement
P. J. Secrest *Off. Dig. Fed. Soc. Paint Technol.*, 33 (May 1961) 583–596
The appearance of a surface colour depends upon the nature of the radiation incident on the surface, the modification of that radiation by the surface, and the response of the visual mechanism to the stimulus finally received. The CIE system of colour specification is based on experimental considerations of these points. The present account is a very simple one and gives the fundamental principles of trichromatic colorimetry. The establishment of the CIE system is explained and definitions are given for CIE primaries, luminance factor, chromaticity, dominant wavelength, and purity. E.COATES

Estimation of Relative Strengths of Dyes Colorimetrically
U. Gugerli *Chimia*, 15 (Jan 1961) 39–52
A discussion in terms of colour physics of the problem of comparing dyed samples when estimating relative strengths of dyes. E.S.

Direct-reading Tomato Colorimeter
R. S. Hunter and J. N. Yeatman
J. Opt. Soc. Amer., 51 (May 1961) 552–554
A description is given of a photoelectric tristimulus instrument, the development of which was requested by the U.S. Department of Agriculture for the colour assessment of tomatoes. A block diagram is presented for the instrument, which is based on the Hunter colour difference meter and computes a final colour value (TC) given by the equation, $\text{TC} = 21\cdot6/(Y_e)^{\frac{1}{2}} - [3\cdot0/(Y_e)^{\frac{1}{2}}] \left[(Y_e - Z_e) (X_e - Y_e) \right]$ in terms of the CIE tristimulus values for source C (X_e, Y_e and Z_e are normalised to 1·0 for the reflectance standard MgO). Earlier work by Yeatman has shown this formula to give highly significant correlation (0·97) with scores assigned by experienced observers. E.COATES

Separation and Quantitative Analysis of Isomers (mainly Dye Intermediates) by Paper Chromatography— II
S. Kitihara, Y. Okuda, M. Takahata, S. Kano, Y. Tanide, and H. Hiyama
Kagaku to Kogyo (Osaka), 34 (1960) 66–75
Chem. Abs., 55 (15 May 1961) 9162
Chromatography on filter paper cut with a triangular end can separate o-, m-, and p-toluidines; o- and p-chloroanilines; 1,5- and 2,6-naphthylaminesulphonic acids mixed with the 1,4-isomer; 1-naphthylamine and 2,6-naphthylaminesulphonic acid mixed with Tobias acid; 1- and 2-naphthylamine; and 1- and 2-naphthol. C.O.C.

Synthetic Food Dyes
A. Carballido and Y. Villanúa

IV— Spectrophotometry of Water-soluble Greens, Blues, and Violets in Acid, Neutral, and Alkaline Media
Anales bromatol (Madrid), 11 (1959) 265–285
Absorption maxima and spectrophotometric curves are given for the permitted Spanish food dyes.

V— Circular Paper Chromatography of the Permitted Spanish Food Dyes
M. T. Valdehita, R. G. Olmedo, and L. Villanúa
Ibid., 287–300
Chem. Abs., 55 (1 May 1961) 8681
C.O.C.

Detection of Added Organic Dyes in Orange Peel
H. Suter and H. Hadorn
Mitt. Gebiete Lebensm. u. Hyg., 51 (1960) 293–303
Chem. Abs., 55 (15 May 1961) 9714
Synthetic dyes could not be separated from naturally occurring carotenoid mixtures by paper chromatography, but absorption chromatography on alumina together with fractional elution led to clear-cut separation of the added dyes which were identified by absorption spectra. C.O.C.

Test Methods for the Water-vapour Permeability and Heat Reflectance of Pigmented Organic Coatings
M. Kronstein, R. M. Sonshine, and J. A. Castronovo
Wet Ground Mica Assoc., Tech. Bull., 1, No. 4 (1960) 6 pp.
Chem. Abs., 55 (1 May 1961) 8885
Describes two test methods for evaluating the influence of differences in the size and shape of the pigment particles in otherwise similar paint systems. The behaviour of free paint films as sealer over water-vapour phases, and their heat reflectance, under conditions of otherwise comparative specular reflectance, were studied. The two methods can

also be used to compare different systems of organic coatings with each other under similar test conditions.

<div align="right">C.O.C.</div>

Complexometric Analytical Methods in the Varnish and Lake Industries
I. Tonini

Deutsche Farben-Zeitschrift, **15** (Apr 1961) 162–165

The mechanisms and uses of the complexometric reagents nitrilotriacetic acid and ethylenediaminetetraacetic acid are discussed, together with the indicators Murexide (C.I. 56085) and Eriochrome Black T (C.I. 14645). The preparation of the reagent is then described, and estimations of Co, Pb, Zn, Mn, Cd, bronzes (Cu and Al), and phosphoric acid are briefly considered. 14 References.

<div align="right">H.H.H.</div>

Chemical Methods for Testing Raw Material in the Wool Industry
H. Zahn *Melliand Textilber..* **42** (Apr 1961) 421–426

Three groups of tests can be distinguished— estimation of foreign bodies, determination of components which are either formed or destroyed during treatment, and indirect methods. The following tests are discussed— determination of fat and soap-like substances, acids, alkalis, cysteic acid by electrophoresis of the wool hydrolysate, lanthionine by paper chromatography of hydrolysate, NH_2 groups formed by action of acid by treatment with fluorodinitrobenzene, hydrolysing and isolating the DNP-amino acids chromatographically (comparison with untreated wool gives the number of extra amino groups). Solubility tests using $0\cdot1$-N NaOH permit detection of alterations in structure without affecting physical properties, e.g. bleaching with peroxide greatly increases solubility and has hardly any effect on tensile strength. Solubility in urea–bisulphite mixtures is directly related to lanthionine content. The test is of interest where alkaline treatment causes formation of lanthionine. The rate of absorption of dye acids is increased by antifelting finish.

<div align="right">W.M.</div>

Identification of Acrilan, Acrilan 16, and Courtelle
J. T. Ball J.S.D.C., **77** (June 1961) 251–252

Identification of Synthetic Fibres Using Optical Methods
P. T. Novoderezhkin

Tekstil. prom., No. 1 (Jan 1961) 43–47

Optical properties, e.g. appearance and colour in polarised light, luminescence under ultraviolet radiation, reflection spectra, have been used to identify various synthetic fibres. For routine identification, preparation of suitable comparison standards is suggested.

<div align="right">L.S.L.</div>

A.A.T.C.C.: 204th Meeting of Technical Committee on Research
Amer. Dyestuff Rep., **50** (3 Apr 1961) P254–P258, P266

The following subcommittees made recommendations which were approved and which will be incorporated in the tests given in the 1961 *AATCC Technical Manual*— Water Resistance of Fabrics (Methods 21, 22, 35, 42, 70A and 70B); Colour Fastness to Washing (Standard method 36–1957 re-affirmed, Tentative method 83–1957 to be Standard method 83–1961 and Tentative method 87–1958 to be Standard method 87–1961). The remaining subcommittees gave progress reports on their activities without submitting recommendations.

<div align="right">S.B.D.</div>

Determination of Steam Fastness under Atmospheric Pressure
Textil-Rund., **16** (May 1961) 266

Method SNV 95 8 80 of the Swiss Standardising Association.

<div align="right">P.B.S.</div>

Determination of Steam Fastness under Elevated Pressure
Textil-Rund., **16** (May 1961) 267

Method SNV 95 8 81 of the Swiss Standardising Association.

<div align="right">P.B.S.</div>

Identification of Antibacterial and Mildewproof Finishes
T. D. Miles

Amer. Dyestuff Rep., **50** (1 May 1961) P325–P328

A.A.T.C.C. Tentative Test Method 94–1961T Category 2. The sample is extracted by the procedure of test method 94, the extract chromatographed, and the chromatograph treated with colour developers. From the colour reactions the materials used in the finishers are identified. Inhibitors are classified as metallic, phenolic, antibiotic, quaternary ammonium compound, or miscellaneous; chromatographic methods are recommended for each class and an analysis typical of one member of each class is given.

<div align="right">P.B.S.</div>

Analysis of Film-forming Finishes in Textiles
B. Norwick

Amer. Dyestuff Rep., **50** (1 May 1961) P329–P335

Selective solvent solubility is used to separate the various materials into groups. Identification then depends largely on the use of infrared spectra, and 18 wavelength-transmittance curves are reproduced. Microchemical additional and confirmatory tests complete the scheme, which is proffered as A.A.T.C.C. Test Method 94 Category 1.

<div align="right">P.B.S.</div>

Analysis of Methods for Determination of Crease-resist Properties of Fabrics
A. M. Shpaer and N. Ya. Tret'yakova

Tekstil. prom., No. 1 (Jan 1961) 47–49

The best conditions for obtaining the angle of recovery (α) are determined. The number of tests required for accurate results depends on angle of recovery. If $\alpha > 55\%$ five measurements are necessary, if $\alpha < 55\%$ ten measurements are required.

<div align="right">L.S.L.</div>

Detection of Nitrocellulose
H. F. Vollmann *J. Oil & Col. Chem. Assocn.,* **44** (May 1961) 308–310

Nitrous acid or N_2O_3 released from nitrocellulose by conc. H_2SO_4 yields a red coloration with an acetic acid soln. of sulphanilic acid and α-naphthylamine (Griess reagent); this is said to give more satisfactory results than the usual diphenylamine method.

<div align="right">J.W.D.</div>

PATENTS

Measuring Quantity of Liquor Absorbed by Textiles
Gebrüder Sucker *BP* 867.327 (Germany 31 July 1956)

An indicator is added to the processing liquor and the amount of indicator taken up by the fibres measured, the measured amount of indicator being used to control automatically the liquor or the machine.

<div align="right">C.O.C.</div>

Method for Determining Hiding Power and its Application as an Aid to Paint Formulation (V p. 459)

Selection of Direct Dyes for Application to Undesulphured Rayon Staple (VIII p. 463)

The Xenon Lamp: An Evaluation of the Tendency of Rubber Compounds to Discolour on Exposure to Light and of PVC Colours to Fade (XIII p. 472)

XV— MISCELLANEOUS

Two-colour Threshold as a Test of Colour Vision
R. M. Boynton and M. Wagner

J. Opt. Soc. Amer., **51** (Apr 1961) 429–440

The method is based on finding the just-visible luminance of a test flash of one colour, seen against the background of another colour. The apparatus, which is described, has been built using two 35-mm projectors with appropriate filters. Neutral wedges were used to vary the luminance of the conditioning field and the test field in discrete steps and rotating sector discs to time the stimuli. Three experimental conditions were employed: (1) steady conditioning field-luminance 20 mL; (2) transient conditioning field-luminance 4 mL; (3) transient conditioning field-luminance 1·25 mL. The tests were carried out on 67 subjects, 21 of whom were red–green defective. Classification of the subjects was obtained as a result of the following tests: (1) Anomaloscope, (2) Colour-matching, (3) Farnsworth-Munsell test, (4) Neutral point, and (5) Dvorine Isochromatic plates. An examination of green on red thresholds plotted against red on green thresholds allows a separation of the normal and the different types of colour-defective observers. The test seems to provide a quantitative assessment of the degree of colour defect and is capable of detecting weak normals.

<div align="right">E.COATES</div>

Chemistry in Scotland and its Pioneer Contributions to Textile Technology

A. Clow   *J. Textile Inst.*, **52** (May 1961) 204–218

The 1961 Mather Lecture. Political events shaped Scotland's economic history, e.g. Treaty of Union 1707 helped linen crofting (bleaching), improvements in which expanded linen manufacture and supplied skills for the rising cotton industry. The invention and manufacture of bleaching powder at Glasgow in 1799 revolutionised the cotton industry. The dyeing of Turkey Red, and manufacture of cudbear and alum were notable Scottish achievements. Discovery of blackband iron-stone in 1800, and the hot-blast furnace in 1828 caused metal manufacture to supersede that of cotton, which further declined as a result of the American Civil War 1861–1866.
                                                                                    R.A.P.

Textile Chemistry in University Teaching and Research

H. Zollinger   *Textil-Rund.*, **16** (May 1961) 247–253

A review of the problems caused by the rapid increase of knowledge in all fields of textile chemistry.   P.B.S.

Sonic and Ultrasonic Vibrations— Applications in Chemical Technology

C. Weimann   *Teintex*, **26** (15 Mar 1961) 190–201;
                                                    (15 Apr 1961) 265–277

Review, discussing theoretical concepts, methods of generating sonic and ultrasonic vibrations, their dual property of dispersing or coagulating aerosols, emulsions, and colloidal dispersions, according to the conditions used, and other applications.   J.C.F.

Steric Effects of *ortho*-Groups on Hydrogen Bond Formation in Phenol Derivatives as Studied by Nuclear Magnetic Resonance

I. Yamaguchi   *Bull. Chem. Soc. Japan*,
                                34 (Mar 1961) 451–452 (*in English*)

The data afforded by 2,6-dichloro- and 2,6-dimethyl-phenol and 2,6-di-t-butyl-*p*-cresol in CCl_4 and acetone (referred to water as an external standard), indicate that the ability of phenol deriv. to form an intermol. hydrogen bond is a linear function of the size of the *ortho*-group (cf. *Ibid.*, **34** (1961) 353).   H.H.H.

Riboflavin as an Electron Donor in Photochemical Reactions

B. Holmström and G. Oster

J. Amer. Chem. Soc., **83** (20 Apr 1961) 1867–1871

Anaerobic photobleaching of riboflavin in the absence of added electron donor first yields leuco deuteroflavin (I) where the two hydrogens added to the aromatic nucleus originate in the side chain. (I) can reduce various substances, e.g. silver ion or dichlorophenol indophenol, being itself oxidised to deuteroflavin. The amount of substance reduced is dependent on the original amount of riboflavin. Analysis of kinetic data indicates that the formation of leuco deuteroflavin involves a long-lived excited (probably triplet) state of riboflavin strongly quenched by the photo-product itself. Formation of leuco deuteroflavin involves acid–base catalysis as shown by increase in quantum yield with increase in buffer concn. Leuco deuteroflavin is autoxidisable to yield the light-sensitive deuteroflavin. Photolysis of this cpd. also operates through a long-lived state but is not retarded by presence of photo-product, namely lumichrome. A reaction scheme is proposed.   F.J.

Spectrophotometric Investigation of Adsorption of Methylene Blue by Highly Disperse Aluminosilicates

L. I. Kul'chitskii

Kolloid. Zhur., **23** (1) (Jan–Feb 1961) 76–85

Adsorption of Methylene Blue (C.I. Basic Blue 9) on highly disperse aluminosilicates involves surface reactions with the formation of organo-mineral salt-like compounds of two kinds. (a) Salt-like hydrates are formed on active basal faces of minerals with layer structure and are characterised by absorption of light in the shortwave region with maximum at 570 mμ, which in time is displaced to 730 mμ. Drying results in the dehydration of these salt-like hydrates with the formation of anhydrous salts. This process is accompanied by the decrease and final disappearance of absorption intensity maxima. (b) Salt-like anhydrous compounds are formed in the regions of broken bonds in crystals of various structures and on amorphous adsorbents, and are characterised by absorption of light in the visible part of the spectrum with maxima at 620, 640, 660, and 680 mμ. This type of compound is stable and the spectrum is not affected by temperature.   I.G.

Instrumentation— Its Success and Delusions

M. I. MacLaurin   *Svensk Papperstidning*,
                                64 (31 May 1961) 394–398 (*in English*)

Paper read at the Swedish Paper Engineers' 1961 meeting, giving examples of how instruments may be developed and installed in a misguided fashion. Continuous control of opacity, consistency, liquid level, curl, substance and moisture are discussed.   R.A.

Radiation Chemistry— A New Possibility for the Chemical Industry

R. J. Coekelbergs

Chem. and Ind., No. 19 (13 May 1961) 612–617

Various sources of radiation energy are reviewed and the available energy is related to the requirements of different types of chemical reaction. Use of fission products offers great possibilities for reactions of small G value; these are confined to a few types yielding products having a large-tonnage sale. Though technological difficulties are at present considerable, such processes may fairly soon become economically attractive. Other radiation sources yield very limited power at high cost and, at the present state of development, appear commercially unattractive.
                                                                                    J.W.D.

Sulphonation and Sulphation

E. E. Gilbert and E. P. Jones

Ind. Eng. Chem., **53** (June 1961) 501–507

Review of work published in 1960 on sulphonation of aliphatic, alicyclic, aromatic, and heterocyclic cpd. and sulphation of alkenes, monohydric alcohols, glycol esters, ethers, and carbohydrates. The following are of particular interest— Surface sulphonation of polyethylene film and use of styrene-4-sulphonate as a co-monomer give polymers of improved dyeability. Sulphonation of benzene, halogenated benzenes, toluene, xylenes, and naphthalene deriv. occupies much of the review. Complete utilisation of sulphuric acid is achieved by addition of $SOCl_2$, which reacts with water formed during sulphonation of aromatic cpd. Sulphur trioxide dissolved in liquid SO_3 is most effective in preparation of sulphonates of long-chain alkyl deriv. of benzene, cyclohexylbenzene, naphthalene etc. Chlorosulphonation of benzene, halogenated benzenes, aniline, and phenol deriv. quinoline, phenothiazine and copper phthalocyanine is described. The last-named cpd. yields a tetrasulphonyl chloride. 167 References.
                                                                                    F.J.

PATENT

Skin-colouring Compositions

N. B. Siccama and W. L. Wirbelauer

USP 2,948,657 (9 Oct 1957)

Even and firmly fixed coloration is developed *in situ* on the skin by precipitating on it a colourless soluble indigoid ester and then exposing to light. Thus a good suntan effect resistant to sea water and soap washing is produced by use of a mixture of C.I. Solubilised Vat Brown 5 (1% by wt.), C.I. Solubilised Vat Blue 5 (0·2), Silicone 555 (Dow) (3), urea (6), Carbitol (0·5), glycerin (0·5), Aerosol OT (0·2), water (10), alcohol (78·6) and ammonia (to bring to pH 8).   C.O.C.

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MISCELLANEOUS

Economy in Dyeing

The Warm (Bicarbonate) Batchwise Dyeing Process for Viscose Rayon.

A simple method has been developed for dyeing viscose rayon with I.C.I. speciality cold-dyeing Procion℗ dyes, using standard dyehouse equipment. This is specially recommended for winch (or jig) dyeing of spun viscose rayon materials and for package dyeing applications.

Dyeings produced by this technique display excellent levelness, penetration and surface appearance and, with certain Procion dyes, — see below — the process yields maximum colour value in full shades.

The dyeing process requires only a degree of control, similar to that used for direct dyes. The dyebath is set with sodium bicarbonate at 50°C. (120°F.), the dyes are added and their exhaustion is controlled by the regulated addition of electrolyte.

Dyes particularly recommended are:—

> Procion Yellow GR
> Procion Yellow 4R
> Procion Brilliant Orange G
> Procion Scarlet G
> Procion Brilliant Red 8B
> Procion Blue 3R
> Procion Brilliant Blue R

PROCION dyes for increased economy,
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Symposium at Zürich   11–14 October 1961

International Federation of Associations of Textile Chemists and Colourists (I.F.A.T.C.C.)

Congress at Noordwijk, Holland   25–27 April 1962